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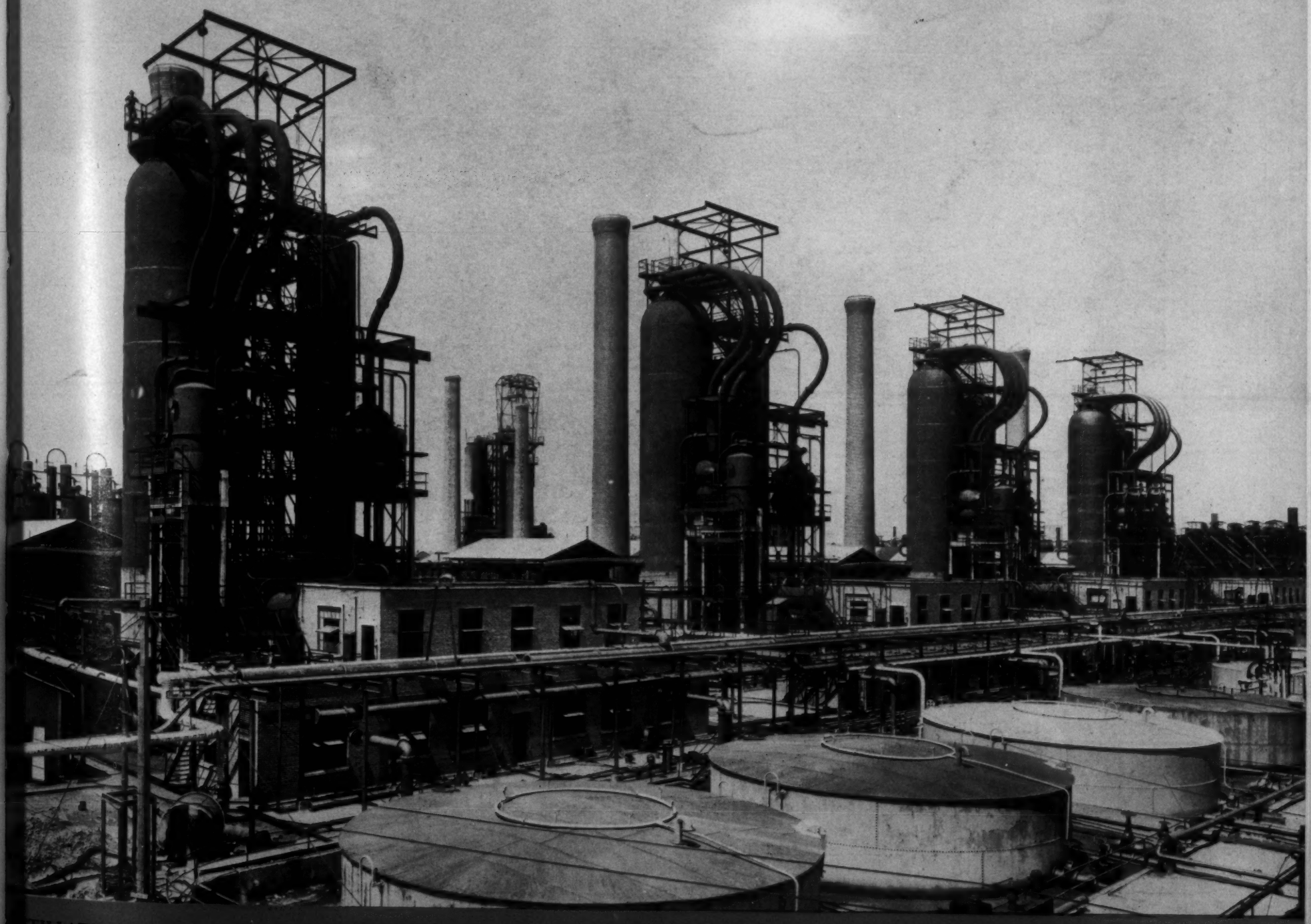
The Magazine of Metallurgical Engineering

INCLUDING
CURRENT METALLURGICAL ABSTRACTS



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VOLUME 4 **JULY 1933** NUMBER 7

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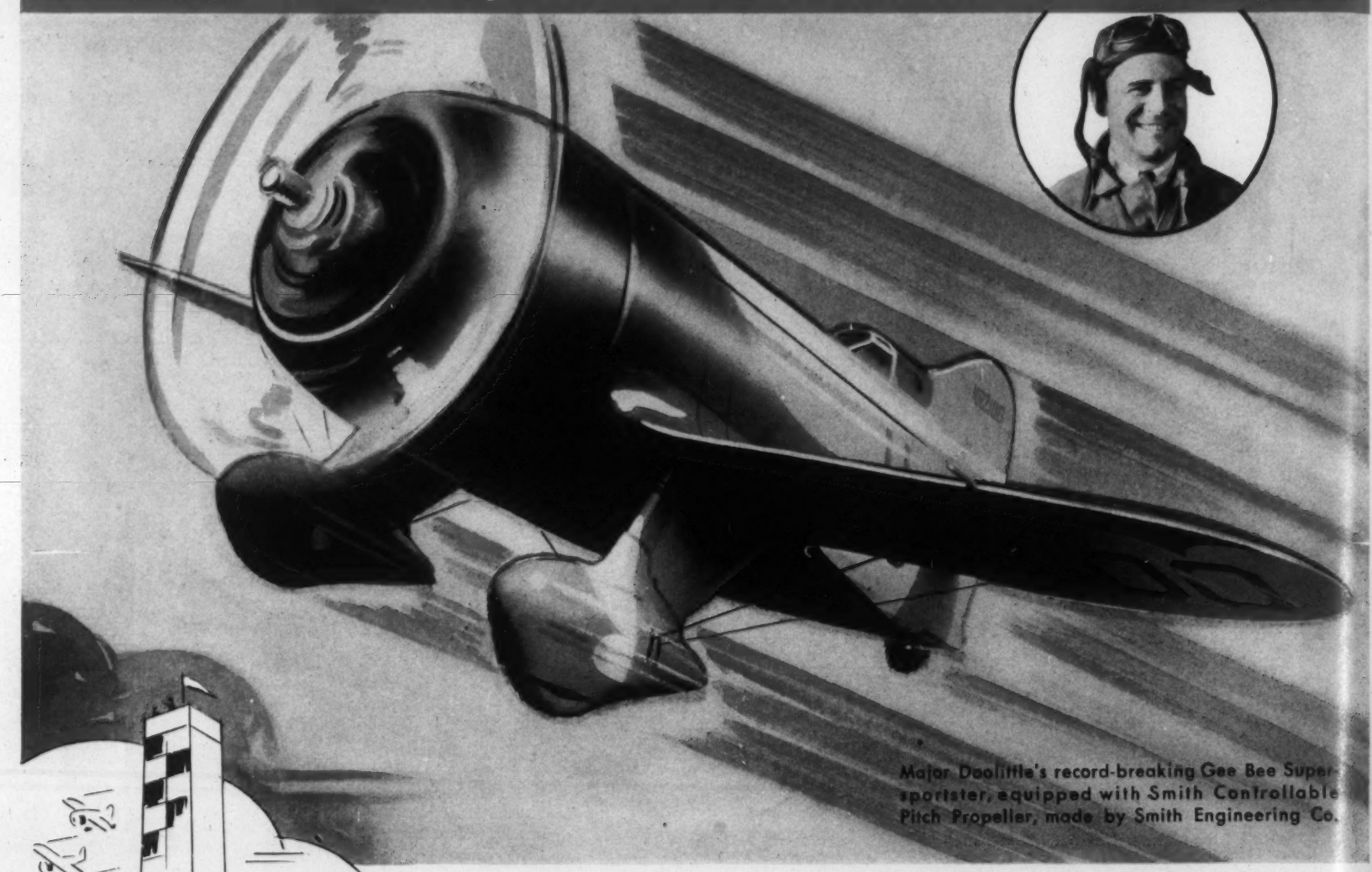
WITH JIMMY DOOLITTLE



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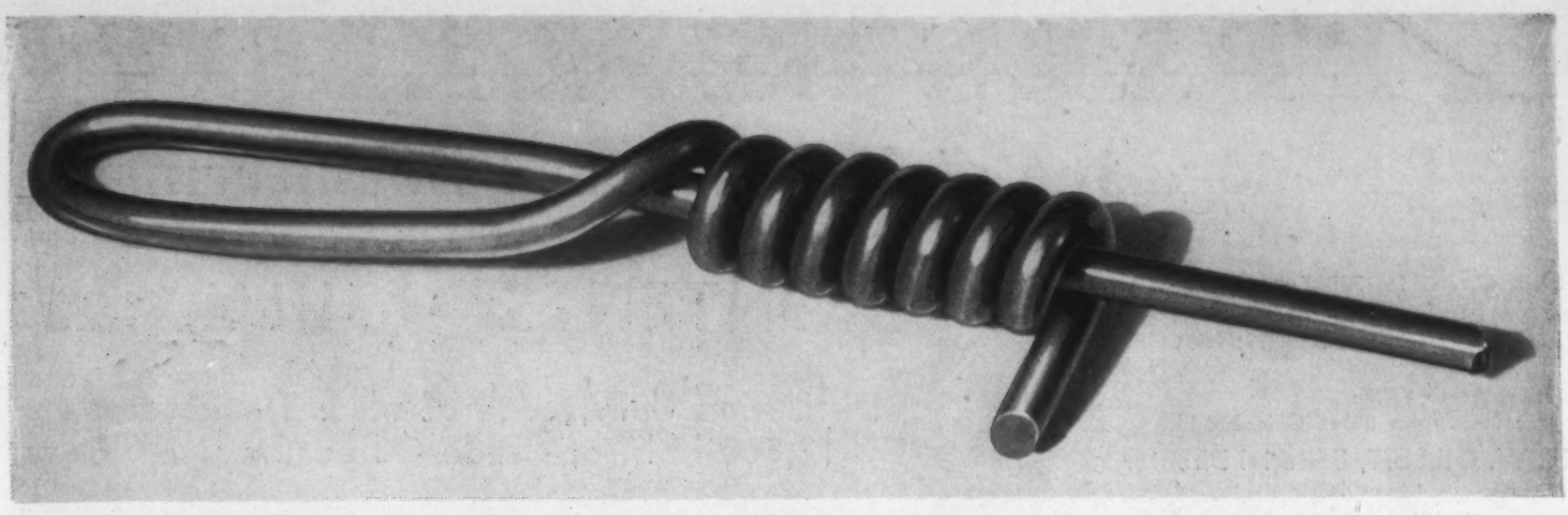

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HIGHLIGHTS

by H. W. GILLETT

Alloys of all kinds.

General surveys of groups of alloys are given by several authors in this month's abstracts. Thews (page MA 205 R5) discusses the Pt alloys: Lambert, all the non-ferrous alloys used in shipbuilding; Saran (page MA 214 R1) light alloy sand castings; while Blumenthal (page MA 205 R9) deals with leaded bronzes, and an anonymous article in Metallbörse (page MA 235 L7) with the effect of nickel in several classes of bronzes. Elliott and co-authors (page MA 208 L6) discuss 23 corrosion resistant alloys for chemical engineering use while Wright and Luger (page MA 211 R7) confine themselves to Cr and NiCr steels for similar use, while Calcott and Olive (page MA 208 L7) write on much the same subject. So does Hildebrandt (page MA 208 L1). Hurst (page MA 208 L2) discusses the corrosion and heat resistant cast irons.

Did you say corrosion?

The "ski's" hold forth on corrosion theories and corrosion testing. See (page MA 207 L3) Prot & Goldowski and Kuczynski & Smialowski, respectively in French and Polish. Cazaud does it in French (page MA 207 L10). Müller & Machu (page MA 209 L3) deal with passivity in German, but, thank God, Kwanji Murata (page MA 209 L6) theorizes on corrosion of iron in English. Two corrosion articles, by Evans & Borgman (page MA 207 L9) and Lee have identical titles, "The Effect of Oxygen Pressure on Corrosion of Steel."

Oxygen and sulphur in steel.

The oxygen content at the time the Si is added in making transformer steel makes a lot of difference, says Bardenheuer (page MA 220 R2).

Sulphur-bearing gases in the open-hearth and how they contaminate the steel are discussed by Eisenstecken & Schulz (page MA 220 R3) on the basis of an investigation in which 3000 gas analyses were made.

The deoxidation of steel, especially by Mn, the slagging effect of MnO on SiO_2, and the effect of CaO in the slagging process are discussed in two English (page MA 220 R9 and page MA 222 L7) and two German, [Krings & Schackmann (page MA 222 L2) von Keil & Czermak (page MA 221 L1)] articles.

DO YOU want to know what metallurgical engineers are saying, the world over? Look in the Current Metallurgical Abstracts. Here are some of the points covered by authors whose articles are abstracted in this issue.

Mill scale ain't what she used to be.

So says Donaldson (page MA 209 L1), it used to be less adherent, which may explain the alleged better behavior of earlier ship plates as to corrosion.

Have you castings to clean?

How to clean castings, pickling, sand and shot blasting and water pressure cleaning are discussed in about the same way in French by Tourneur and in German by Prachtl (page MA 223 L7).

Welding cement? No!

"Welding technique in the cement industry." No, they are not welding cement, but the steel appliances used in the industry (page MA 220 L3).

Don't jar a structure during welding says Etter (page MA 220 L6).

They figure costs down to a hair in the Navy, for Wallin and Shade (page MA 220 L8) say the Navy saved $14,-846.40 in welding an auxiliary vessel, as well as saving 17% in weight over rivited construction.

Amid all the articles on welding steel, there is one lone article on lead burning (page MA 220 L9).

Silver art castings.

Iron, steel, bronze and aluminum castings are frequently discussed, but silver castings are not everyday matters to most foundrymen. However, there's an article on Practical Silver Art Casting (page MA 230 R10). That's real silver, while the next abstract below it deals with "German Silver" art castings.

Don't nickel plate over cadmium.

For a durable electroplated coating, start with a clean base, say Steen and Leonhardt (page MA 215 L7). Don't nickel plate over a cadmium coat says Thews (page MA 215 R1), while Justh (page MA 215 R3) does not care for either Cd or Zn under coats for Cr. Beutel & Kutzelnigg (page MA 215 R3) say that a Cd coat on brass turns yellow through diffusion unless the Cd is deposited from a fluosilicate bath. Hentschel (page MA 215 R9) advised using more H_2SO_4 in Cr plating baths than is usually advocated.

Machining with diamonds.

Diamond tools for machining are said by Meyer (page MA 222 R10) to average 1000 hrs. service before needing refinishing, while Winter says they will cut commutator brushes at a speed of over 15,000 ft./min.

Case hardening and malleabilization and precipitation hardening.

Rough checking of case depth for control purposes—note the grinding sparks—suggests Thebault (page MA 219 L4).

The how and why of malleabilization—White & Schniedewind (page MA 219 L10).

Is the effect of hydrogen in palladium analogous to that of precipitation hardening? Feussner (page MA 219 L10) suggests that it may be.

How about sand cores?

Breitenstein (page MA 230 R3) claims that warm core sand coated with oil, poured into the core box, needs no ramming, but will set or "freeze," the operation taking less time than ramming.

Notches, surface defects and decarburized surfaces.

Five articles (page MA 214 R5) discuss the effect of notches and surface discontinuities. Thum and Oschatz show how additional notches can be used to decrease the damage due to an unavoidable notch. Hankins and Becker (page MA 214 L4) also discuss the effect of decarburized surfaces.

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Editorial Comment

Quench in Oil

SO READ THE INSTRUCTIONS for heat-treating many steels. There is probably no other factor in heat-treatment less standardized or less closely regulated by the average heat treater than the quenching oil. Yet it is a highly important factor, for it may happen that if the oil is one of too slow a quenching speed or one whose quenching speed is adversely affected by a slight rise in temperature, it may not properly harden a steel whose critical quenching rate is relatively high.

The composition of the steel and the time and temperature of heating for quenching are very closely controlled, yet the steel is quenched in a medium of which most metallurgists can usually say only that it is "oil," perhaps qualifying that by a trade name or brand number that conveys no scientific information. Yet if the metallurgist tries to use machine oil or some other "just oil" that he picks up around the plant, he soon wishes he had used a quenching oil. Thus the compounders and sellers of quenching oils have done the metallurgist a service, for they supply something he needs but cannot himself specify in scientific terms. He is, however, rather much at their mercy, and it sometimes happens that the selling of quenching oils is accompanied by much alleged information and many claims for superiority that carry more than a trace of bull.

Quenching oils of the earliest days were animal or vegetable oils, whale, fish, linseed, cottonseed, rape seed, etc., but cost (and odor in the case of the first two) brought a change to mineral base oils. The present oils are evidently mineral oils, perhaps blended, and often with a few percent of a fatty oil. There is very little on record either as to what bases and compounding additions are used, or why. Nor is it easy to set down just what properties are required. A high flash point is necessary to avoid fire hazard; a high cooling rate, and one that does not change rapidly with temperature, are generally wanted; and a low enough viscosity to avoid too much "drag-out" is an advantage, as is stability of the oil. But there are other factors involved. French[1] found that a mixture of two mineral oils, used commercially for its high quenching rate, did show a high rate on ordinary oxidized steel, but did not if the steel were nickel or chromium plated. Something analogous to the wetting power of a cutting oil for finish turning or of a compounded lubricating oil seems to be involved.

Empirical investigations made by compounders of quenching oils have resulted in serviceable materials, for which the user pays plenty for the "know-how" of compounding them and for the brand name. It would appear that so important a tool in the metallurgist's kit should be better understood, and be capable of purchase on specification and evaluation on test.

It is odd that the oil companies, who do considerable research on many relatively minor uses for oil that would appear far less important in industrial life than that of quenching oils, pay little attention to this field and are content to sell the mineral base to middlemen instead of actively seeking a direct market, though they sell direct when someone tells them the grade he wants. The initiative is left to the metallurgist.

Probably the reason is that the average oil company knows of metallurgy only as it affects its own production, as in cracking stills, and has little metallurgical talent on its staff, while the average metallurgist is not at all conversant with petroleum products. It is odd, too, that the metallurgist has paid so little attention to the possibilities of improving, and lowering the cost of, quenching oils. Some large users of quenching oils do buy raw materials direct, and make up their own mixtures. But they don't publish the specifications on which they purchase the oils, though it would seem nothing to be secretive about. It would be interesting to know how closely the specifications of different users agree and the details of the investigations that have led them to their conclusions, whether the conclusions agree or differ.

French and co-workers at the Bureau of Standards paid some attention to the question in their broad survey of the quenching process, and they, as well as others who have dealt solely with the evaluation of critical quenching rates, have provided methods by which the quenching power of oils might be quantitatively evaluated. Krekeler and Rapatz[2] have given a brief discussion of the quenching oil problem. But the work on oils has been pretty much of a side issue, for, though commercial oils of unknown composition were compared with a few mineral and fatty oils of definable and reproducible properties, no attempt seems to have been made to study the question from the point of view of compounding a quenching oil of better properties, or one that can be made up by the user from available raw materials. Quenching oils appear, to the average user, still to be in the patent medicine era of secrecy and hokum.

Then, too, a quenching medium, to bridge the gap of quenching speeds between water and oil, is much desired. There have been some commercial attempts to use emulsions, but their practicality is in doubt and a little work by Hamill[3] led him to believe them to be so difficult to produce and maintain at a known quenching speed, that he turned to the study of other possible intermediates, such as glycerine and water glass.

Probably the oil industry has physical and colloid chemists who know their emulsions and would not take as pessimistic a view as the metallurgist would.

The whole field appears to be one in which the oil producer and the heat-treater ought to get together for research. Neither one will go far without the knowledge and the point of view of the other. Together they ought to be able to make quenching oil something definable by specifications upon which all can agree rather than a Lydia Pinkham type of product.—H. W. GILLETT

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[1]H. J. French. *Transactions American Society for Steel Treating*, Vol. 17, 1930, pages 646-727; 798-888.

[2]K. Krekeler & F. Rapatz. *Archiv für das Eisenhüttenwesen*, Vol. 5, 1931, pages 173-176.

[3]T. E. Hamill. *Bureau of Standards Journal of Research*, Vol. 7, 1931, pages 555-571.

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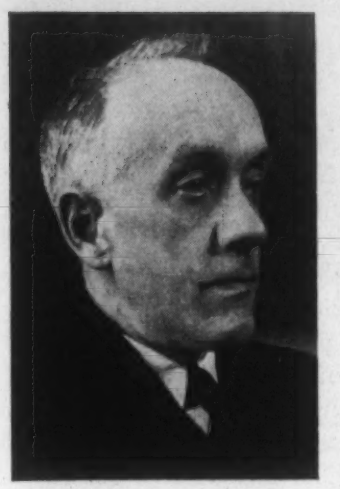


Obtaining Reliable Values for CREEP of METALS at HIGH TEMPERATURES

by H. W. Gillett* and H. C. Cross*

THE designers of heat-treating furnaces and the like, of steam power plant equipment, of oil-cracking stills, vessels, and pipe lines, of steam turbines, and many other kinds of equipment have to use heat resisting alloys. Hence, they must make an intelligent selection from among those available. They must know or decide what amount of deformation can be allowed without harmful effects during the useful life of the equipment. If reliable test data are available for the material they plan to use, they can then design so that the unit loads will not produce more than the allowable deformation at the temperature of service. The sections necessary for strength cannot in some cases be used due to limitations of thermal conductivity or cost. The engineer must then either use material of greater strength or reduce the temperature of service.

In steam engineering and in oil-refining, the needs for economical operation are driving the designer to higher and higher temperatures and pressures. Progress is retarded by lack of better alloys for high temperature service and by lack of accurate knowledge of the properties of those available.

Design has been largely on the basis of service experience, but the designer is anxious to put it on the basis of experimentally determined load-carrying ability at high temperatures for long times.

The furnace engineer now generally phrases his design loads in terms of 1% creep per 10,000 hours, the oil and steam designers, of 1% per 100,000 hours, and the turbine designer, of 1% per 1,000,000 hours.**

SOME of the most difficult engineering problems of today have to do with the applications of metals and alloys at extreme temperatures and pressures. The metallurgist has a clear duty to develop better alloys for high temperature use, and to collect and disseminate information that will allow the engineer to use the materials now available right up to the permissible limit and to prevent him from overstepping it. The securing of reliable data on the high temperature properties of metals and alloys is extremely important.

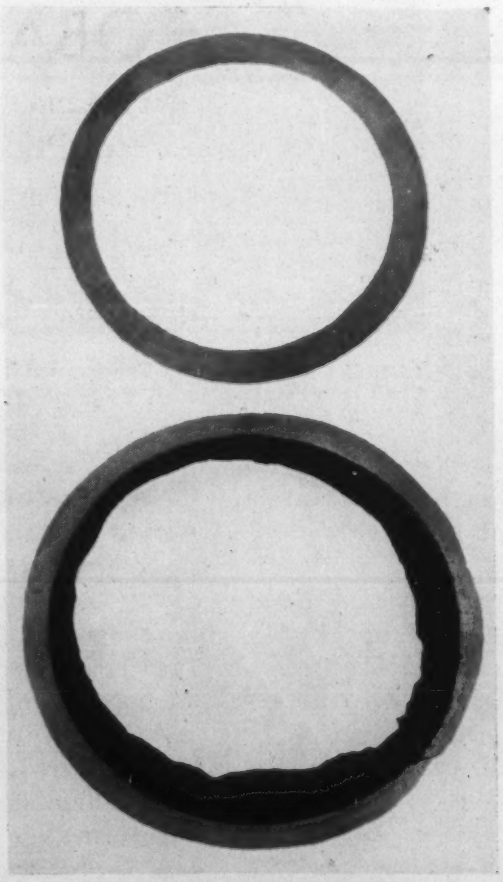


Fig. 1. Sections cut from a cracking still tube after removal from service, obtained through the courtesy of an oil refiner. The tube is of 5% Cr steel without other alloy additions. The smaller section was taken from an unfired zone while the larger section, with coke deposit, was taken from the furnace zone. The larger section was originally of the same dimensions as the former but creep has taken place due to effect of the pressure at the higher temperatures. It was removed when just at the point of failure. just in time to prevent explosion and fire. Oil at 900° F., 750 lbs./in.². Metal temperature reaches 1200° F.

Since few if any laboratory creep tests have actually been run over 5,000 hours, and 2,000 hour runs are none too common, and since the exact determination of a final rate of creep of 1% per 100,000 hours requires more than usual care, test figures so phrased are the result of much extrapolation. It is more logical to express them as 1%, 0.1% and 0.01% per 10,000 hours, and hence, not to imply so much as to behavior beyond the actual period of test.*** It would be still better to express them as 0.1%, 0.01% and 0.001% creep in the second 1,000 hours of a 2,000 hour creep test, thus avoiding extrapolation entirely and showing that a real and not an "accelerated" creep test had been made.

Operating engineers are usually very hazy on the exact loads and temperatures really existing in service. They usually give the designing engineer nothing better than a fairly good guess. They can be excused for this in some degree, for precision measurements are not easy to obtain in the field. Much more attention should be paid to this and the designing engineer should demand more accurate operating data. At any rate, he does not wish to superimpose a guess from the metallurgical laboratory as to the properties of a new alloy upon the operator's guess as to operating conditions and then use the combination for the calculation of design stresses, since he is likely either to waste much metal or to invite failure.

Fig. 1 illustrates the importance of creep in engineering design for high temperature service.

The engineer believes that the testing laboratory is the place for precise measurements and he demands trustworthy data on high temperature creep properties. So far, he does not feel much reliance in the laboratory figures, and his hesitation to take them at face value is justified when he sends the same material to two laboratories

*Battelle Memorial Institute.

**For details on the above subjects see:

Symposium on Effect of Temperature on the Properties of Metals, A.S.T.M.-A.S.M.E., 1931.

L. W. Spring & H. W. Maack, Metallurgy of Valves, Fittings, and Pipe Materials. *Metals & Alloys*, Vol. 1, April, 1930, pages 446-449.

J. J. Kanter & L. W. Spring. Present Aspects of Materials for High Temperature Service from the Creep Test Standpoint. *Metals & Alloys*, Vol. 1, December, 1930, pages 880-882.

J. B. Romer. Metallurgy and the Power Industry. *Metals & Alloys*, Vol. 1, December, 1930, pages 868-870.

***This phraseology is used in the new A.S.T.M.-A.S.M.E. Creep Code. See Appendix to Joint High Temperature Committee Report Proceedings A.S.T.M. 1933. Pt. 2.

of long experience in creep testing and high reputation and gets results that indicate perhaps 100% difference in final creep rate. Such data, unpublished, but passing by word of mouth from one designer to another, do not increase their faith in laboratory results, but do not diminish their wish for trustworthy and concordant data.

Creep at the rate of 0.1% per 10,000 hours, is a ten-millionth of an inch per inch per hour. This is a precision measurement even at room temperature. To make it at 1200°, 1400° F. or higher, and with a degree of accuracy that will justify extrapolation from probably not more than a 3 month's test, at best, to service of ten years or more, is a real task.

The development of proper creep test methods is of such fundamental importance that it has been a major task of the Joint Research Committee on Effect of Temperature on the Properties of Metals, of the American Society for Testing Materials and the American Society of Mechanical Engineers. This Committee and its subcommittees comprise nearly all the American workers on creep of metals.

In June, this Committee submitted† a Tentative Test Code for Long-Time High Temperature (Creep) Tensile Tests based on the collected experience of its members and on all facts available in the literature.

This Code imposes requirements for precision that make obsolete the equipment and methods of a good many of the laboratories that have not been taking the necessary precautions to secure accurate results. We may therefore expect less difficulty from discordant data in the future than in the past. Indeed, while the Code appears to be a drastic one from the point of view of those not appreciating the necessity of stringent requirements, some of the Committee members feel that future revisions of the Code will be such as to impose still more rigid requirements.

The writers share this view since, in ten years of creep experience, they have progressively, from year to year, found it necessary to go to still greater refinements in creep testing. A description of some of these refinements and an expression of the ideas that led up to their adoption, may be of interest to other workers in this field.

The problem may be phrased, "How may measurements of the needed precision be made, at the lowest over-all cost? What equipment and methods will give it?"

The pioneers in creep testing obtained much useful information with crude equipment and methods, but the time is past when any but precise methods can be usefully applied when design values are sought. Nevertheless, one must take care not to add to the complication and expense of equipment and methods unless they really add needed precision.

Preliminary Tests

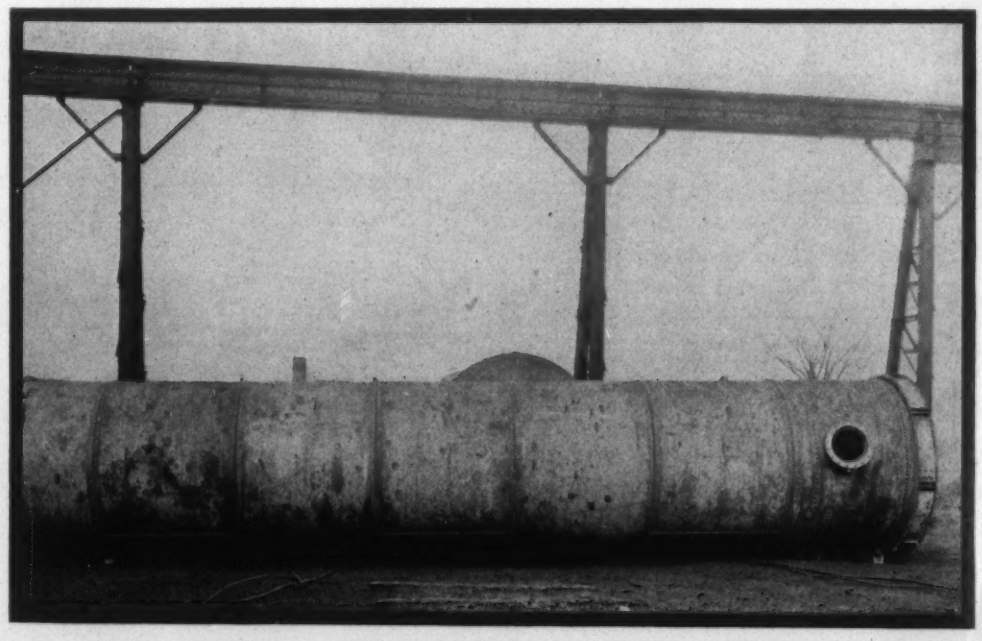
Creep tests are so expensive in equipment, electric power, and time of experienced men, and so long-drawn out at best, that time spent in getting a good foundation for an intelligent guess at the load that will produce the desired final rate of creep is well spent. Ordinary high temperature short-time tensile tests are of some value provided the creep curves are known for another material of the same class with similar short-time test properties. Running the short-time type of test, holding the specimen at temperature and at a given load for the desired time and measuring permanent set after it cools to room temperature seems as useful and more convenient than attempting to make a precise proportional limit test.‡

Simple cantilever loading or dead weight compression tests at high temperatures, carried out for quite a long period on a group of materials or on the same material with different heat-treatments often serve to select the material or treatment for further study by creep test.

Scott[1] has described an interesting permanent set test on a bowed specimen, and Mochel[2] has used a compression test on a spiral spring in such range-finding work.

Preliminary Survey of Material for Test

Picking the proper load will greatly shorten the time for an adequate creep test. Every creep test engineer knows this, but some do not yet realize the importance of avoiding a difficulty that may scrap the results of a series of otherwise correct tests. Unless the samples for a series of creep tests on a given material are really alike and representative of the material that is to go into service, the results will not be trustworthy. The material should be examined for hardness, microstructure, soundness (preferably by X-ray for all cast material) and often for magnetic properties, before the creep test, to show up any variations. McVetty[3] has discussed this fully.



15-18% Chromium Iron Tower, used in Production of Nitric Acid, manufactured by Downingtown Iron Works. (Courtesy Electrometallurgical Sales Corp.)

It is cheaper, too, to determine by separate tests that on account of too rapid oxidation or lack of structural stability a given alloy is useless for service at a given temperature than to find this out in the course of creep tests. Shrinkage or other dimensional changes due to temperature alone and not to load, which would render the creep observations inaccurate, should be determined separately.[4]

Types of Creep Tests

Coming to the creep test itself, one has first to decide

†Appendix II to Joint High Temperature Committee Report, *Preprint* No. 23, American Society for Testing Materials, 1933.

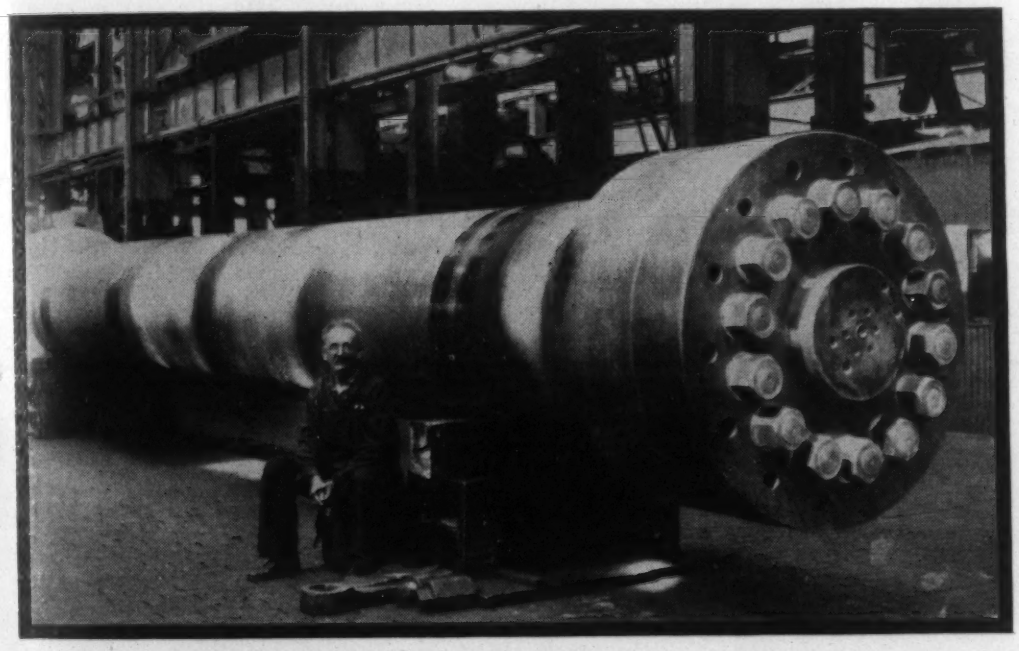
‡The Joint High Temperature Committee has also put out a Tentative Code for Short-time High Temperature Tensile Tests (Appendix I to the 1933 Report) and has shown (Appendix IV) that proportional limit tests at high temperatures are not precise enough to justify the attempt to make them.

what type of creep test he will adopt. Everett[5] has made out a good case for the torsion test and it is hoped that more data on it will be forthcoming, but the difficulties in preparation, from much of the material one wishes to study, of the tubular specimens required to best utilize the torsion method seem at present to outweigh its advantages for any but special purposes. The great bulk of data on record are tensile data and one hesitates to forego opportunities for direct comparison.

Type of Specimen

Having decided on a tensile test, the dimensions of the specimen must be fixed. Some work has been done with small diameter specimens and two smaller sizes are allowed by the Code when a standard specimen cannot be obtained, but these appear to be so greatly affected by oxidation, when testing oxidizable alloys in air, that for reliable data on creep, the standard 0.505″ diameter specimen, which is generally adopted, should be used wherever possible. To apply the necessary loads for the lower temperature tests, a specimen of this size requires a rugged equipment and the use of a lever system or its equivalent instead of dead loading.

The length of the test section is governed by the methods to be used for measurement of extension and for



Forged Chromium-Vanadium Vessel for Ammonia Synthesis manufactured by The Midvale Company. (Courtesy Vanadium Corp. of America.)

attainment of uniformity of temperature over the section. These factors will be discussed below. While some very long specimens are in use, most laboratories use a 2″ gage length and few use over 4″. By adjusting other factors so that the 2″ gage length will suffice, test material will be conserved.

Loading the Specimen

There are so-called creep testing devices in which the load or temperature is automatically reduced till creep, within the precision of measurement adopted, comes to a standstill. Useful as these may be for preliminary tests or for special purposes, they do not give the information as to the rate of creep at one load and one temperature wanted by most designing engineers. Hence, we need equipment that will apply loads up to say 75,000 lbs./in.^{2} to a 0.505″ diameter bar with the loading accuracy of a good tensile testing machine. Kanter and Spring[6] use rugged lever arms and knife edges made by a testing machine manufacturer and load the bar vertically, to avoid friction in the cable and pulley system required with a horizontal test bar, and to make possible the use of a platinum strip for measurement of extension.

Most laboratories use some such type of equipment. Since the equipment is usually constructed by the user, it is an advantage to be able to buy the loading system on the open market, so in choosing creep equipment for Battelle Memorial Institute, we adopted Kanter and Spring's[6] design.

The lever arm ratios must be carefully checked and the weights properly calibrated, but these precautions introduce no difficulties. Precautions must be taken to avoid twisting the lever arms on the knife edges and to apply the load gently. An adjustable screw is used as a stop to prevent the lever arm falling too far in case a specimen breaks in the machine, but using this to apply the load after the specimen is up to temperature tends to displace the lever arms on the knife edges, so we support the lever arm by a small hydraulic jack and let this down gradually when applying the load.

Norton and Romer[7] comment that vibration of the building is a limiting factor in the precision of their outfit, so, while our laboratory has gratifying freedom from vibration, since all heavy moving equipment, motors, rolling mill, hammer, etc., are mounted on independent foundations in the basement, we have mounted our creep units on ½″ of "Keldur" to absorb vibration.

The machine should apply truly axial loading to the specimen. A ball and socket joint is, of course, provided, but that alone does not sufficiently insure axial loading. McVetty[3] has emphasized the importance of axial loading. Very high grade machining should be done on both adapters and specimens so that the threads of both are true and parallel to the reduced section of the test specimen. We use extra long threaded grips on the specimens (Fig. 2) and advise the reading of extensions on two sides of the specimen so as to reveal any lack of axiality of loading.

With such equipment and precautions the load measurement in creep testing should be as accurate as it is in good tension testing. However, it is not accuracy of loading but accuracy of temperature control that is difficult of attainment.

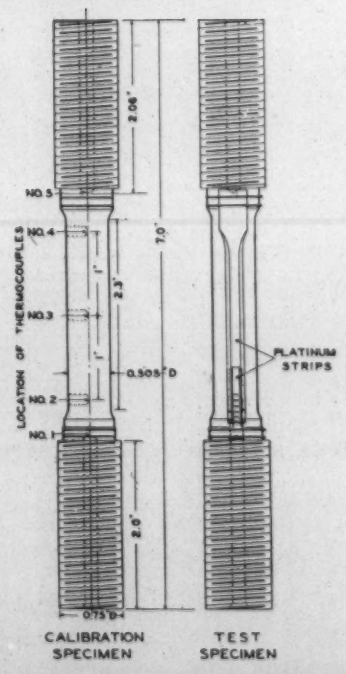


Fig. 2. Types of temperature calibration and creep test specimens used at Battelle Memorial Institute. Note the platinum strips used for measurement of creep.

Temperature Control

Several laboratories have tried putting several specimens in one furnace, with the idea that on comparative tests of different materials, or of different loads on the same material, all at one temperature, the comparison would be better because several specimens would be tested at one time and any differences in temperature control in consecutive runs would thus be avoided. Coffin and

Swisher[8] report good results with one furnace for several specimens, by dint of many precautions, but most of the laboratories whose early outfits took several specimens have gone to one specimen per furnace in later installations, finding that this made for convenience in many ways. We have therefore avoided using more than one specimen per furnace. The Code advocates one specimen per furnace.

How close must the temperature control be for a given degree of precision? In the temperature range where creep resistance begins to fall rapidly as temperature rises, i.e., the range about which the designing engineer is most concerned, an increase in temperature of 20° F. will often double the rate of creep. In a particular case of a low alloy steel that has been quite thoroughly studied, the final creep rate for a certain load at 700° F. is 0.1% per 10,000 hrs. and 0.2% at 720° F. If the thermocouple calibration is off 5° F., if the temperature controller "hunts," ± 5° F. over a range of 695°-705° F. holding the temperature at 700° F. for ¾ of the time and at 695° F. and 705° F. each for ⅛ of the time, if room temperature changes affect the controller outfit so as to cause a + 5° F. error, and if the temperature distribution over the 2″ gage length runs from 700° F. in the middle inch up to 720° F. at the ends, it can be calculated that the *observed* creep would be at the rate of 0.23 per 10,000 hrs. while the true creep is but 0.1%. That is, the test would indicate that for design in which 0.1% creep was the limit, the useful life would be 4½ years instead of 10 years. Were the errors all on the low side, 0.05% creep might be indicated or a useful life of 20 years.

Putting the errors into terms of stress values, what appears to be the true load for 0.1% creep is 20,000 lbs./in.², in the low alloy steel referred to, while accumulated errors all on the high side would indicate it to be 15,000 lbs./in.², and all on the low side, 25,000 lbs./in.² Two laboratories comparing the same material might readily report these two widely varying values and would only be certain to agree more closely when the temperature errors had been reduced well below those assumed in the computations referred to above. Other materials at other temperatures may be even more affected by a slight difference in temperature.

Taking up the individual sources of error, it is not easy to calibrate a thermocouple and maintain it through a 2,000 hr. run so as to be sure that its indicated temperature is correct to less than 5° F. We have repeatedly satisfied ourselves by tedious calibration that no change as great as 2° F. has occurred in the couples during long runs, but feel that such calibration cannot be omitted on important tests. Methods for calibration are well known and need not be discussed here, but it is obvious that very careful calibration is required and that the calibration should be checked after a creep test as well as before it. The Code specifies that the temperature measuring equipment shall be accurate to ± 3° up to and including 1200°, ± 5° up to and including 1600° F. and ± 10° F. above that.

In our units two thermocouples are located in each creep test specimen during the tests. They are inserted axially through holes drilled in the adapters and specimens, one in the bottom shoulder of the test specimen and the other in the top shoulder. The bottom or control thermocouple is connected through a switchboard to an automatic temperature controller. The upper thermocouple is also connected through a switchboard to a recorder which draws a continuous record and shows how evenly the temperature has been maintained. To read the temperatures more precisely than can be done from the recorder charts, a portable potentiometer is plugged into the switchboard so as to read the temperature of either the upper or lower thermocouples or both.

The best location and method of shielding of the thermocouples in the test specimen is still in dispute. The Joint High Temperature Committee of the A.S.T.M. and A.S.M.E., through its sub-committee on analysis and correlation of data of short-time tests,[9] favored putting the couple within the specimen itself, in the filleted portion close to the test length, while McVetty[3] and Tapsell[10] prefer to hold the thermocouple on the outside of the specimen and shield it from direct furnace radiation. We have followed the Joint Committee recommendations of 1927. The Tentative Code proposed by the Joint Committee allows either location.

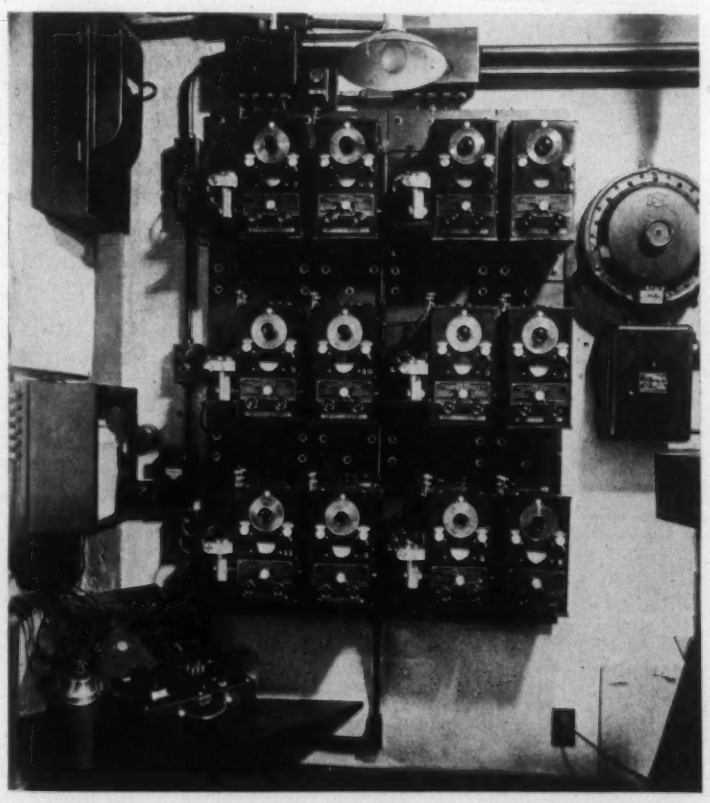


Fig. 3. Equipment used for controlling and recording temperatures in creep tests at Battelle Memorial Institute. Note the plug board to which all thermocouples are run and the portable precision potentiometer used for checking the temperatures.

Some laboratories run several creep furnaces from one control on the plea of economy in first cost, balancing the various furnaces by rheostat adjustment. Our experience of some years ago in attempting to do this convinces us that it is poor economy and each furnace should have its own individual controller. In multiple operation, anything that goes wrong with the controller or any furnace in the circuit tends to spoil the whole set of tests.

The Code advises a separate control for each furnace. Fig. 3 shows the equipment used for controlling and recording temperatures in the creep tests at Battelle Memorial Institute.

The temperature of the room in which creep test equipment is located should also be maintained as near constant as possible, since the equilibrium of the temperature gradient in the furnaces and adapters is dependent on this factor. The Code specifies not over 10° F. variation in room temperature.

Room temperature varies not only with the weather but also from the heat thrown off by the furnaces that heat the test specimens. This amount of heat varies with the number of units that are operating and their respective temperatures.

We have therefore installed a fan which exhausts the warm air near the ceiling of the room to the outside atmosphere and at the same time draws in cooler air from

the corridor through louvers in the door. This fan is controlled by a Mercoid thermostat located just above the creep test equipment. The thermostat is set for 85° F., a temperature slightly above the lowest possible of maintainance during the summer. This greatly minimizes room temperature changes but does not entirely remove them.

A source of error that is only taken care of in some of the most modern control devices concerns the dry cells by which the controller circuits are balanced. Tests showed our controllers to have very satisfactory cold junction compensation if the controllers were balanced and the compensation checked at the same temperature. However, if the controllers were balanced at one temperature and then the cold junction compensation checked at temperatures above and below that, it was found that an error ranging from 3° to 5° occurred as the result of a change in instrument temperature of about 10° F. Greater changes than 10° did not produce much additional error. This error was due to the change of e.m.f. of the dry cells as caused by the instrument temperature, i.e., room temperature change.

ond cycle, variation at the center of the gage length of a dummy calibration bar is less than 1° F. and at the ends of the gage length less than 2° F. as measured by a precision potentiometer.

Most laboratories have to depend on the power company to maintain the correct voltage. The recorder charts will show if any voltage variation too great for the controller to handle has occurred. We have not had serious difficulty on this score or on continuity of current supply.

At rare intervals when the current must be cut off for line repairs or changes, the power company gives notice and care is taken to insure that the proper temperatures are again obtained when the power is again available and before the specimens are reloaded.

Temperature Uniformity Over the Gage Length

The above discussion relates to holding the temperature of the control couple constant. Even though this temperature be constant, there will be an error if the test length is not all at the same temperature, but has a temperature gradient. In order to get any approach to a

Table I. Temperature Uniformity of Creep Test Specimens as Affected by Furnace Liners of Different Metals

Location of Thermocouple[1]	Low Alloy Steel					"18-8" Steel		
	Furnace Without Lining	Furnace Lined with Copper Sheet .020" Thick	Furnace Lined with Nickel Sheet .031" Thick	Furnace Lined with Silver Sheet .020" Thick Greatest Variation[2]	Furnace Lined with Silver Sheet .020" Thick Least Variation[2]	Furnace Lined with Silver Sheet .020" Thick Greatest Variation[2]	Furnace Lined with Silver Sheet .020" Thick Least Variation[2]	Furnace Lined with Nickel and Chromium Plated Copper Sheet .040" Thick
800°F.								
#5 Top Key	844	800	810	802	800	810	803	802
#4 Top Gage Length	820	799	796	800	800	804	801	800
#3 Center Gage Length	800*	800	800*	800	800	800	800	800
#2 Bottom Gage Length	808	802	804	800	800	800	800	799
#1 Bottom Key	831	804	817	802	802	806	803	803
1000°F.								
#5	1058	1004	1021	1006	1004	1010	1006	1005
#4	1025	1000	998	1000	1000	1003	1000	1000
#3	1000*	1000	1000*	1000	1000	1000	1000	1000
#2	1012	1002	1009	1002	1000	1001	998	1000
#1	1037	1011	1028	1008	1000	1010	1000	1008
1200°F.								
#5	1282	1211	1242	1207	1204	1210	1211	1213
#4	1237	1200	1205	1200	1198	1200	1200	1200
#3	1200*	1200	1200*	1200	1200	1200*	1200	1200
#2	1224	1205	1209	1205	1200	1194	1200	1202
#1	1261	1217	1242	1215	1203	1207	1212	1209

*Temperature variation over gage length greater than allowed by Tentative Code.
[1]Thermocouple locations as noted in Fig. 2.
[2]Greatest and least variations noted in calibrating 12 different furnaces.

To minimize this error, we have placed all dry cells used for controller balancing in an insulated thermostated box which is itself located in the thermostated creep test room but maintained at a slightly higher temperature.

Some controls are being built with automatic balancing to the actual room temperature at short intervals and others avoid the use of dry cells. The dry cell error, when present, is often overlooked and if it can be present in the outfit used, it needs consideration in any creep test unit.

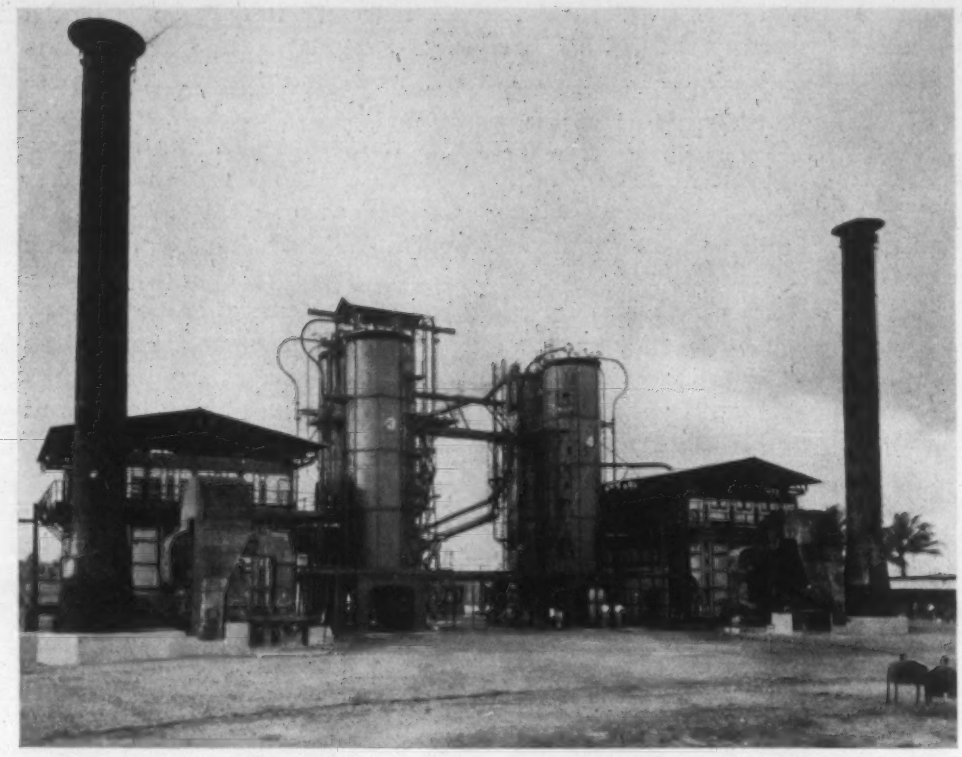
The "hunting" of the controller will cause some temperature fluctuation as it throws the current on and off. This is a potent cause of error in some installations and seems to have been successfully avoided in the practice of Kanter and Spring[6] and also by Coffin and Swisher.[8]

Kanter and Spring[6] have fully described their method, which we have followed. They ran the major portion of the current required through the furnace constantly and the remainder was switched "on and off" by the controller. "Anticipatory Control" was obtained by locating in the same insulator with the control thermocouple, a loop of chromel wire connected in series with the "on and off" portion of the furnace current. With fairly constant voltage supply and the controller operating on a 10 sec-

reasonably small temperature gradient, gap-wound furnaces are essential, providing more heat to the ends of the specimen to compensate for that lost through the grips. The end windings may be in series and be given the correct resistance by trial and error, may be in parallel and separately adjusted by external resistance, or may be on separate circuits. The longer the test length, and the higher the test temperature, the greater the difficulty in getting temperature uniformity. A change from one set of grips to another of different thermal conductivity or test specimens of different conductivity will also affect the gradient. Any change in dimensions or method of insulation of the furnace will affect it. Extensometers that have parts sticking out of the furnace drain off heat, and greatly complicate the problem of test length uniformity.

Each furnace must be calibrated by a dummy bar of the same conductivity as the material to be tested, with the same grips as will be used, and at all temperatures at which it is to be used. The requirements of most laboratories on uniformity of temperature prior to the adoption of the Code do not appear severe enough since lack of uniformity over the gage length is one of the most prevalent sources of error.

Oddly enough, Tapsell[10] does not state National Phys-



Dubbs Cracking Units equipped with Foster Wheeler Tubular Oil Heaters and Plate Type Air Preheaters. Oil is heated to temperatures up to 950° F. in these units. (Courtesy Foster Wheeler Corp.)

ical Laboratory practice in this regard. Coffin and Swisher[8] claim only 2° F. variation even over the long gage length they use, by means of separate windings and external resistance. Norton and Romer[7] originally allowed 20° F., Clark and White[11] state theirs as 10° F. Kanter and Spring[6] give 10°-15° F. We find the Kanter and Spring design to give the best results of any we have tried, and in general, we stick to their exact recommendations as to furnace dimensions and winding. Even so, some furnaces are not as good as others, and it requires moral courage to go to the expense of discarding furnaces that are not quite up to standard on calibration.

Barr and Bardgett,[12] Prulière[13] and others have used sleeves of 18-8, copper or silver to secure better temperature uniformity. We tried lining a furnace that showed a very poor gradient, and which would have had to be discarded, with copper, nickel, or silver sheet, with the results shown in the following table. The uniformity can, of course, be improved still further, in the cases where entire uniformity had not been obtained by the metal sleeve alone, by the use of auxiliary windings and external resistances. It is desirable that the "key" couples in the fillets, used for control, give readings close to that of the gage length.

The nickel sheet, though thicker than the others, did not give satisfactory results, its thermal conductivity apparently being too low.

The bare copper oxidized too much, but silver was very satisfactory up to 1100° and usable at 1200° F., though at the latter temperature there is some volatilization of silver. For high temperatures we have used various plated coatings for silver and copper. With great care and the use of properly gap wound furnaces, suitable metal sleeves and separate windings and corrective resistance where necessary, it should be possible to come within a total variation of 5° over the gage length, at least up to 1600° F.

The Code specifies that the gage length shall show no variation over 5° for temperatures up to and including 1200° F., 10° up to and including 1600° F., and 20° above that. This is a requirement that very few creep outfits in use have met in the past, but it is one of the points that some of the Committee members, as well as the writers, believe should, and probably will, be made even more stringent in the future.

While current practice uses thermocouples for both control and measurement of temperature, Hatfield and co-workers[14] have reported good results from the use of a platinum resistance thermometer control. This has the very great theoretical advantage that the control is actuated by changes in resistance of platinum and this is a function of the absolute temperature, so that no cold junction troubles exist. Such controls might avoid the necessity of thermostating the room, provided that the extensometer used is not of a type that is affected by room temperature changes. We are trying out furnaces so controlled and so far find them very promising but we need longer experience before we could definitely decide in favor of the resistance control.

Whatever type of furnace or control be used, it is necessary that the furnace and specimen be brought to temperature equilibrium before applying the load and this must be done slowly, requiring 24 to 48 hours, depending on the initial current used. The higher the heating rate, the more carefully the furnace must be watched, and in any event the final heating must be done slowly.

So much for temperature difficulties. Given a certain limit of error due to lack of perfect temperature control and distribution, it is obvious that a method of measurement of extension of the test piece is required that is of comparable precision.

Measurement of Elongation

A low alloy steel may expand 0.000007 in./in./ °F. Then a temperature variation of 3½° F. arising from any combination of causes, will produce a change in length of 0.000024 in./in. A creep rate of 0.1% per 10,000 hours is 0.0000024 in./in./day. Hence, it would take 10 days actual creep at this rate to produce the same length change as a 3½° F. change in temperature. An extensometer that can be reliably read to 0.00005 in. on a 2″ test length will show up 0.000025 in./in. The plotted creep curve will contain the combined errors from lack of temperature control and actual lack of precision in reading. If we have a total error of 0.00005 in./in. from both sources, from the theory of probable errors, it will take nine consecutive daily readings to reduce the error of the curve to 0.00002 in./in. In other words a test must go on for at least 200 hours after the true rate of creep has fallen to 0.1% per 10,000 hours for the readings to prove that this rate is being maintained and not exceeded. Unless the temperature of the specimen is decidedly less than 3½° F. off from the supposed temperature, the expansion or contraction error will nullify the advantage of a greater precision in length measurement.

Suitable precision in measurement of length changes may be sought in various ways. The crude methods in which no extensometer measurements were taken on the specimen itself, but over-all changes in length of specimen plus grips were measured, have long been obsolete because of the effect of room temperature changes on the grips. One may increase the length of the test specimen and use a rather crude extensometer, as do Coffin and Swisher[8] or may use a shorter specimen and more sensitive extensometer.

According to Tapsell,[10] Bailey of the Metropolitan Vickers Company, London, uses an 8″ gage length and a Marten's type extensometer. The N.P.L., he says, uses

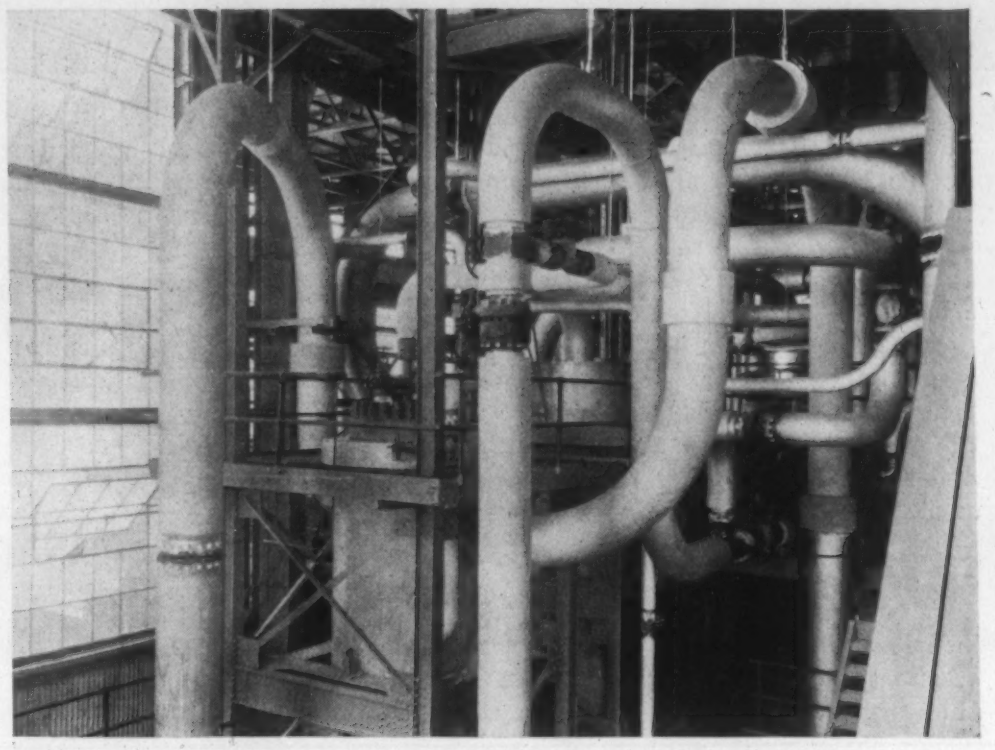
the Marten's type on a 2″ gage length, reading to 0.000002 in./in., but in much work uses a less sensitive extensometer which measures to 0.00003 in./in. McVetty[3] uses a Marten's type extensometer and a 3″ gage length. Clark and White[11] describe, and Clark, White and Thomassen[15] refer to a Bauschinger type extensometer reading to 0.000003 in./in. on a 2″ gage length used at the University of Michigan. Kanter and Spring[6] use an optical extensometer sensitive to 0.000025 in./in. on a 2″ gage length and Norton and Romer[7] use a similar device of similar sensitivity.

In choosing an extensometer, we considered it useless to try for a sensitivity out of proportion to the precision of temperature control and uniformity. We have used the Marten's type extensometer in short time high temperature tests, in one of its more serviceable forms,[16] and were not impressed with its suitability for day in and day out creep work. The disadvantages of lack of ruggedness, of harmfully affecting the temperature distribution in the specimen by draining heat from one end of the furnace, and of having parts extending into the air so that the gradient in and expansion of the legs are functions of room temperature, all count heavily against it. The optical micrometer method of Kanter and Spring[6] seemed preferable on the score of ruggedness and of introducing nothing to extend out from the furnace.

To compare the day in and day out performance of the different types of extensometers there were available much data published by Kanter and Spring, curves taken with the Martens, kindly supplied by McVetty, and published curves from Coffin and Swisher.[8] These data on which all observed readings were plotted, were for tests under conditions such that there was either no creep or a very small and steady rate of creep. Enlarging these photographically to the same scale, they could be compared for deviations of individual readings from the curves. Comparing the three different sets of available curves, no apparent advantage was shown by any one extensometer method. As Kanter and Spring's data were for shorter gage lengths than the others, and their specimen best lends itself to attainment of temperature uniformity over the gage length, the optical extensometer method was chosen.

The optical method must be used in such fashion that the length change can be measured within the field of the objective of the micrometer telescope without moving the telescope itself from a fixed position. Norton and Romer[7] use two telescopes properly spaced, one merely to reference the position of the other, and sight on reference marks on platinum beads welded onto the shoulders of the specimen. Earlier attempts by others to use sharp shoulders of the specimen itself for sighting upon have had to be abandoned because of errors due to oxidation and scaling.

Kanter and Spring[6] use a platinum strip attached to the upper shoulder and measure the gap between that and a platinum wire set into a groove on the lower shoulder. The strip method requires that the specimen be vertical. We have followed this plan, but for materials that show relatively large initial extension, we have modified it by use of two strips as shown in Fig. 2, on each of which are finely ruled cross-marks. One may start with two certain cross-marks and if the extension is so great that they can no longer be used, conversion is made to a set closer spaced, thus fully utilizing the resolving power of the telescope objective.



Steam to Steam Reheater at Deepwater Station of Houston Lighting & Power Co. Steam is received from the boiler at a pressure of 1292 lbs./in.² and a temperature of 825° F. This steam reheats 350 lbs. steam to a final temperature of 715° F. (Courtesy Foster-Wheeler Corp.)

Such methods involve the differential expansion of platinum and of the steel tested, and ignore any stretch in the fillets outside the 2″ gage length. Calculation and experience show that these errors are too minute to cause concern. The utilization of supersensitive length measurements should be deferred till super-constant temperature control and uniformity are achieved.

Some laboratories have used test bar fillets of small radius to avoid any fillet error, but doubtless fall into a greater error due to stress concentration just outside the fillet. When specimens of smaller diameter than the standard must be used, this must be watched very carefully.

In using the optical extensometer, it has been customary to remove the mica covering of the windows through which one sights, when making a reading. Fearing errors due to cooling from this source, we have made the windows of clear quartz, ground flat on both sides so the telescope can be sighted through them without removing them.

There is a possible personal error in the use of the optical method, since the observer must always select the same places on the marks for measurement. To reduce this error, readings of the specimens under test are made daily by two experienced observers and the readings of both are made a part of the permanent records. Both do not see the reference marks the same, for the readings usually differ slightly but the rate of elongation is usually the same. When preparing curves, the curves for both observers are plotted and then an average curve drawn through them.

Plotting of Data

The curves should be plotted to a scale that is sufficiently open. We use 1″ per 100 hours, and ½″ for 0.1% or 0.01% elongation, according to whether the criterion is 1% or 0.1% creep in 10,000 hours. A line showing the slope for this criterion is drawn above the creep curve for convenience in comparison. All readings should be shown on the curves and no report should be made containing assembled plots of final slopes without also presenting the primary curves. The design engineer should be forced to read the data from curves which will show him how long the tests were run and how much individual readings varied. He then has only himself to

blame if he overestimates the accuracy of the data. There is too much publication of derived data only, unsupported by primary data. The engineer might well be forced to make his own logarithmic plots, if he happens to believe that logarithmic presentation is desirable,[4] for then he will more fully realize that such curves are of more value for interpolation than for extrapolation.

Presenting the plots of direct data also avoids confusion as to whether the value reported refers to total creep, including initial extension, or merely to the final rate of creep, since both methods of reporting are used and engineers desire both types of information. It also tells the user of the data, whether the work was done by the accepted method of determining creep at one load and temperature or by the doubtful methods of step-up or step-down loading.

Time Required for Running Creep Tests

How long to run a creep test depends on how much information is available as to the strain hardening range of the type of material under test, so that one may be able to predict how the creep rate may change after very prolonged loading, and on what the criterion is as an acceptable final rate of creep. When the creep rate for a given specimen is so high as to be far above the acceptable criterion, nothing is gained by running it after that is established. For loadings that result in a rate close to the criterion, we find, in general, that a *minimum* of 500 hours for 1% per 10,000 hours and 1,000 hours for 1% per 100,000 hours is necessary before the trend of the curve is sufficiently established, and in really precise work, twice these periods is none too much.

We see no use at all in attempting to get design values from any short "time-yield" test or similar short-cut method. Such figures may be helpful in picking the loads for real creep tests but deserve only to be considered as "sighting shots."

The creep Code states that the following requirements must be met in order for creep tests to be reportable as standard.

Maximum Temperature Fluctuation °F.	Minimum Sensitivity of Elongation Measurement Parts per Million of Gage Length	Minimum Time of Test-Hours	Data Reportable as Creep Rate in % per 10,000 hrs.
1	4	2,000	0.01
2	20	1,000	0.1
5	100	500	1.0
5	500	250	10.0

The Code is not clear as to just what is meant by "fluctuation." A "hunting" of the controller which might change the temperature of the specimen as a whole over a range of 2° F. about the average temperature, would obviously have no worse effect than having half of the test length 2° F. above or below the nominal temperature, and not so bad an effect as having the whole test length 5° F. off the nominal from faulty thermocouple calibration, or from a cold junction compensation error. Within limits, one may make up for lack of sensitivity of elongation measurement by running the test a longer time, but not so for a temperature error. With the Code allowance of ± 3° in thermocouple accuracy and 5° in temperature uniformity over the gage length at 1200° F. it would appear that if the term "fluctuation" is taken as meaning "variation from all causes," as it probably should be, data that are properly reportable in terms of 1% per 10,000 hrs., are scarce.

In our own work at 1200° F., for example, with a temperature gradient over the gage length of not over 3°, controller hunting under 2°, thermocouples checked within 2°, room temperature controlled, compensation errors eliminated, extensometer readings to 0.000025 inches per inch, and runs of 1,500 to 2,000 hrs., we feel that a creep rate of 1% per 10,000 hrs. is accurately revealed, and that figures for 0.1% per 10,000 are probably fairly reliable.

Detection of Deterioration of Metals at High Temperatures

The creep test is only one factor in the appraisal of an alloy for high temperature service. Even though the creep values are satisfactory, other changes in properties, not discernible in the creep test itself, may make the material of little value. These changes may not be shown on mere heating of a specimen not loaded but may require load for their development. Hence, those specimens which do not creep at a greater rate than could be allowed in design should be given a postmortem examination to show whether or not the data may be taken at face value. Signs of lack of structural stability, such as changes in hardness, magnetic properties, microstructure, resistance to impact, etc., should be carefully sought on specimens after creep testing in order to prevent unwarranted extrapolation to longer periods than the actual test period and to warn of any detectable harmful effect that has begun during the test. Cast specimens should be turned down in order to detect blowholes that might have been present to affect the results.

Conclusions

With due attention to the examination of specimens for uniformity before testing, proper study of them after creep testing, and the use of the precautions in temperature control and uniformity and of the other precautions that experience has pointed out, rather reliable creep test data can be provided for rates not less than 0.1% per 10,000 hours at temperatures that are not too high. With all precautions that it is practical to apply, the determination of such rates as 0.1% per 10,000 hours does not as yet appear reliable, such rates requiring too much extrapolation to justify complete credence.

Neglect of the necessary precautions, such as are summarized in the Code, and discussed above, is fatal. Poor, misleading data in this field are much worse than no data at all.

Since it is obviously necessary to make specific tests on each material to get data for design purposes, no one laboratory can itself find time to test all the materials in which it is interested. It is therefore imperative that creep testing methods be refined and unified so that the results of different laboratories, and of different test units in the same laboratory, will give strictly comparable results. Rough methods will not serve. Even so-called "routine" creep tests have to be made with all the precautions of the research laboratory to produce useful results. This is a field in which eternal vigilance is the price of the accuracy that the design engineer is demanding and will continue to demand.

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(Continued on page 104)

A Few Problems in Non-ferrous Castings*

by Dr. Georg Masing**

1. SHRINKAGE AND PIPING

The soundness and serviceability of castings depends upon many factors, among which the factors controlling shrinkage and piping are very important.

In order to discuss the factors controlling shrinking and piping it will be necessary to define precisely a few of the terms as they are used here.

By "linear shrinkage" we shall mean the percentage change in length between mold and casting. By "contraction in solidification" we mean the percentage change in true volume on melting or freezing. By "linear contraction in the solid state," we shall mean the percentage change in length suffered by the casting in cooling from the temperature of final freezing to room temperature.

Metal poured into a mold cannot contract in length until a solid skin has been formed by the chilling action of the mold, a skin strong enough to retain the weight of the molten metal in its interior. The lowering of linear shrinkage by alloying can often be explained, to a first approximation at least, simply by the temperature lowering at the end of solidification by the alloying metal.

The general problem is not so simple. Linear shrinkage depends upon the velocity of solidification, for example upon whether the metal is chill cast or sand cast.

It may be seen from Table 1, after Bauer and Beck that with the exception of nickel the shrinkage in the chill cast condition is larger than that in the sand cast condition. Shrinkage must, to some extent, depend on the crystal structure of the metal, because the largest differences between the sand cast and the chill cast state are encountered in metals of lower crystalline symmetry than the cubic. The reason for this difference is at present unknown. Apart from the possible gas evolution during solidification, there may be a force of crystallization, which causes the crystals to grow in certain directions and thus to exert a pressure on the skeleton of the solidified metal already formed. Immediately after its formation, this skeleton may give way to the pressure.

Table 2 shows data for some alloys, all of which crystallize in the cubic system. Nevertheless, there are great variations in the percentage differences between the shrinkages in the chill cast and the sand cast conditions. These figures lead to the conclusion, that shrinkage is largely influenced by some factors other than the natural constants of the metals.

Shrinkage is primarily caused by linear temperature contraction in the solid state, but depends also on other factors. We have already mentioned two such factors, the intrinsic pressure due to gas evolution and the force of crystallization. The strength of the crystal layer on the surface may not suffice to withstand the inner pressure developed in one way or another and thus the outer shape of the ingot may be slightly deformed. If the source of the inner pressure is the force of crystallization, voids need not necessarily be produced within the metals, as there may be a suffi-

DR. MASING here discusses several of the important problems in the production, structure and properties of non-ferrous castings. The problems of segregation—both normal and inverse, of grain size and structure, of gas evolution during freezing, of gassing for purposes of purification, of deoxidation, all problems upon which Dr. Masing has worked for some years and upon which he is an authority. The theoretical and practical aspects of these problems are of great importance, particularly since much research remains to be done. This article should be very useful to the plant man as a summary of the present status of the field. The present section includes a discussion of shrinkage and piping and of segregation; the concluding section, dealing with gases, deoxidation, and structure, will be published in the next issue.

Table 1. Shrinkage of metals in the sand cast and the chill cast state.

Material	as sand cast in %	as chill cast in %
Ni	1.986	1.681
Cu	1.850	2.042
Al	1.600	1.895
Zn	1.096	1.611
Pb	.750	.942
Sn	.225	.695

*Condensed from a lecture delivered at the Carnegie Institute of Technology.

**Metallurgist, Research Laboratory, Siemens-werke, Berlin-Siemensstadt, Germany.

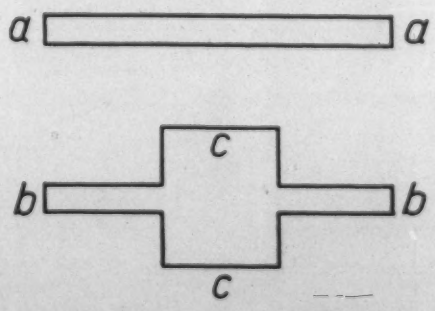


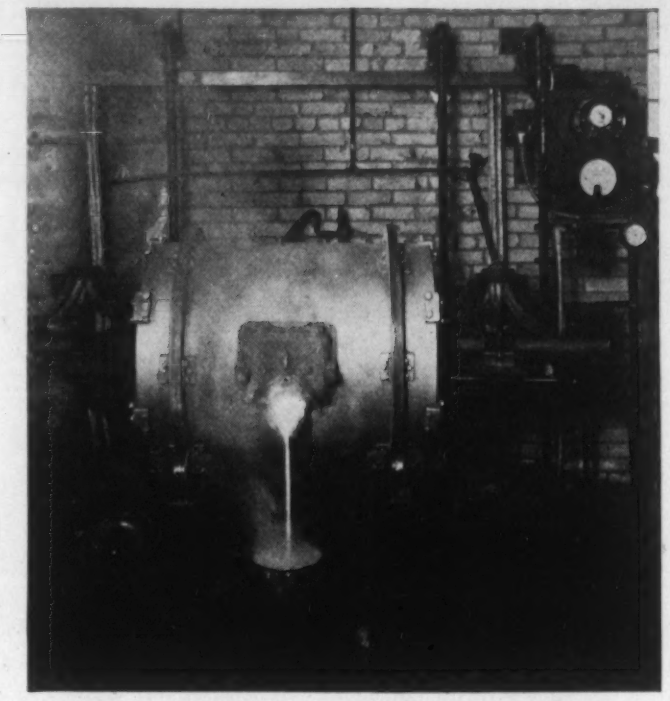
Fig. 1. Influence of the Casting Shape on Linear Shrinkage.

Table 2. Shrinkage of some alloys in the sand cast and the chill cast state.

Material Name	Composition %	as sand cast	as chill cast
Duraluminum	Cu 3.5, Mg 0.5, Mn 0.5	1.335	1.516
Brass	Cu 72, Zn 28	1.763	1.866
"	Cu 57, Zn 41, P 1	1.408	1.938
"	Cu 58, Zn 39.5, Pb 2.5	1.675	2.225
Cu-Ni	Cu 80, Ni 20	2.083	2.292
Aluminum bronze	Cu 90, Al 10	1.400	2.208

cient supply of liquid to fill them.

If shrinkage depends on the rate of solidification, it must also depend on the shape of the casting. Consider two castings in a sand mold. (Fig. 1) In the case of rod a, the outer layer solidifies at once and there is therefore a definite shortening of the rod, corresponding to a definite shrinkage value. But in the case of rod b, the thicker part c will solidify much later than the thin ends. Shrinkage cannot begin before c is solidified since there is no solid connection between the two ends b; these two ends will undergo a certain temperature contraction, without appreciably changing the length of the whole rod, and only later, when c is also solidified, will the true shrinkage of the rod be started.



Tapping a Rocking Electric Furnace.
(Courtesy Detroit Electric Furnace Co.)

Shrinkage is therefore not a purely physical problem, but also a technological one, depending on the form and the distribution of mass in the casting.

The same thing is more clearly visualized in the case of piping. The physical cause of piping is the volume contraction of the metal during solidification. From a physical point of view, the factors of piping are therefore known, when the specific volume figures are given for the melt and for the crystals at the crystallization temperature. Piping is, however, a purely technological problem which demands that we know the part of the metal body and the geometrical form in which the volume contraction will occur. The well-known means of avoiding or influencing piping consist in a regulation of the heat flow during solidification in different directions.

2. SEGREGATION

Segregation is caused by a difference in composition of the crystals formed first and the remaining mother liquid and thus originates during solidification. This condition occurs in almost all commercial alloys.

The simplest case of segregation is that caused by differences in specific weight of the melt and the crystals. The crystals have a tendency to rise or fall according to their specific weight. This kind of segregation requires a great difference between the specific weights of the crystals and the melt. Only alloys without extensive solid solution formation will therefore show a marked specific weight segregation. Such segregation requires time, and therefore is found in the case of slow solidification. The simple specific weight segregation is seldom so pronounced as to be of any consequence in practice.

The second kind of segregation is caused by the fact that crystals tend to grow at points where their nuclei have been formed. Nuclei occur at the most rapidly cooled points, usually at the surface of the castings, whence the crystals grow into the inner part of the metal body, while the mother liquid accumulates in the core of the casting. In this case segregation is determined by the direction and amount of heat flow in the solidifying metal.

The direction of the concentration change caused by the segregation is determined by the constitutional diagram of the alloy. In the alloy x, Fig. 2, the crystal separated from the melt contains more of the component A than the melt. The parts of the casting solidifying first must therefore contain an excess of A, while the core will contain an excess of B.

This kind of segregation is called direct or normal. There are many cases where it occurs, especially in alloys which do not form solid solutions extensively, and in alloys, where the second component B is not really dissolved but merely suspended in the melt. Thus sulphur and phosphorus segregate normally in iron and steel, oxygen segregates normally in copper. Surprisingly normal segregation is found in alloys of gold and copper, which show complete solubility in the solid state at solidus temperatures.

Normal segregation is a direct result of the type of constitutional diagram. It is to be expected in all cases, where it is not obstructed by other factors. Even when the factors interfere, there are some traces of normal segregation.

Curiously enough segregation is frequently opposite in sign to normal segregation. This effect is called inverse segregation.

Again consider the diagram in Fig. 2. Although the crystals separated from the melt are richer in A than the mother liquid, an excess of B over the average concen-

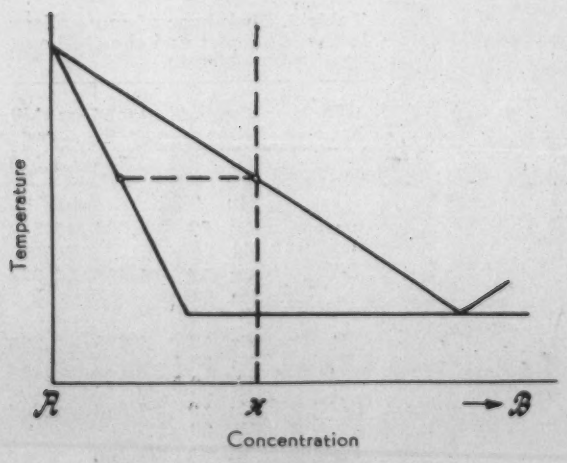
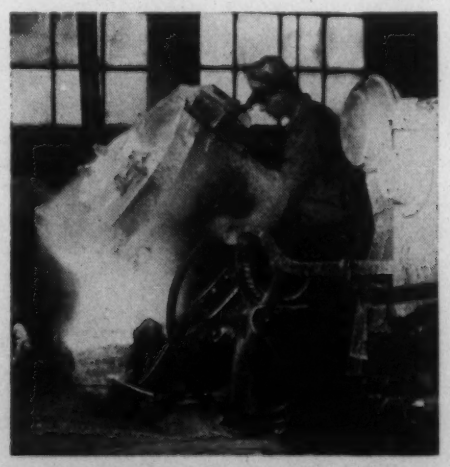


Fig. 2.



Tapping Red Brass Valve Alloys.
(Courtesy Ajax Electric Furnace Corp.)

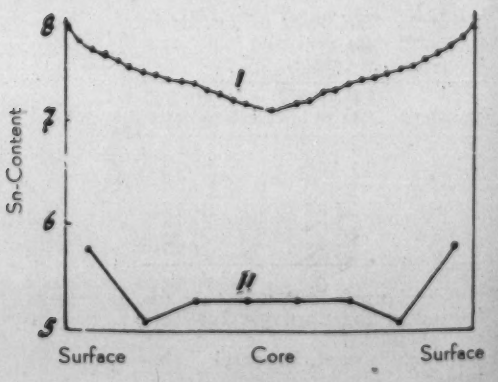


Fig. 3.

tration of the alloy is found in the parts crystallizing first that is, the surface of the castings, whereas an excess of A is found in the core, where accumulation of the mother liquid should be expected.

This paradox is difficult to explain, though many attempts have been made. Several explanations adduced have proved to be false and further thought is called for.

Bronze with various contents of tin and other metals such as lead or zinc shows inverse segregation. Fig. 3, curve I, shows the distribution of tin in a chill cast bronze containing about 7.5% Sn. It will be seen, that the tin concentration rises continuously from the center to the outer surface of the casting, in contrast with what might be expected from the constitutional diagram.

In this case the solidification of the small ingot in the cold thick metallic mold was very rapid. By heating the mold to 300°-400° C. or by pouring small ingots in sand it is possible to avoid segregation almost completely. But in the still slower solidification, as with large castings in dry sand molds, inverse segregation occurs again. If we plot the amount of inverse segregation against the time of solidification we obtain Fig. 4. At very high cooling velocities, when the whole metal specimen solidifies nearly at once, no segregation whatever should occur. With very low cooling velocities, at which the solidification process takes place under rigorous equilibrium conditions, the inverse segregation will again disappear.

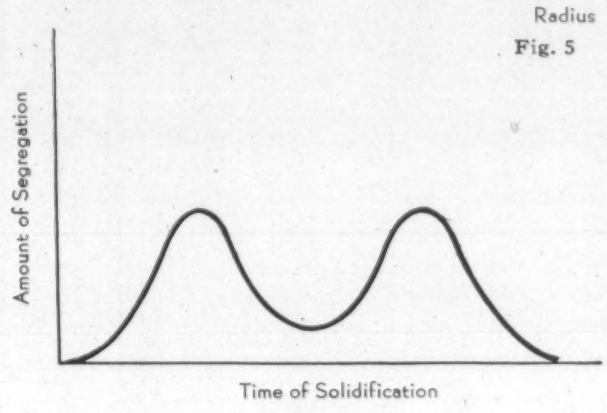


Fig. 4

The dependence of inverse segregation on cooling velocity indicates that there must be two types, one noticed at high velocities and another at low velocities. To clarify the theory it is essential that effects occurring only at low or only at high cooling velocities be not confused but clearly distinguished.

It is probable that inverse segregation at low velocities is caused by gases dissolved in the liquid alloy and evolved during solidification. Drops of liquid appear at the surface of solidifying bronze castings, just before reaching complete solidification. At the top surface of the casting, there are, in the worst cases, whole streams of the mother liquid rich in tin which come out of the interior. This effect is always associated with porosity in the inner parts of the ingot. The force driving the mother liquid out of the core of the ingot can only be the hydrostatic gas pressure generated by evolution from the liquid alloy during solidification.

Every one dealing with bronze sand castings, is familiar with this phenomenon of "sweating." The solidifying process begins quite normally. At the top of an ingot one can note the advance of the border of the crystalized part of the metal. At the same time the surface of the molten metal sinks and piping begins. Then, when the whole surface seems to be crystallized, a drop of a liquid metal suddenly appears at one or more points of the surface and becomes a liquid stream that solidifies at once in the air, thus forming little eruptive peaks. Obviously the gases absorbed by the melt are enriched in the mother liquid; as soon as their concentration exceeds the saturation limit, they are suddenly evolved, develop a pressure and press the mother liquid through the intercrystalline channels still filled with the melt.

The concentration distribution of tin in a sand cast ingot of about 5¼% Sn as shown in curve II of Fig. 3 is in accordance with this description. We notice signs of direct segregation in the inner parts of the ingot, the tin content sinking a little from the core to the outer parts of the casting. Only the surface layer shows a higher tin content, obviously obtained by a comparatively quick transfer of the mother liquid from the inner part to the surface at the end of the solidification process.

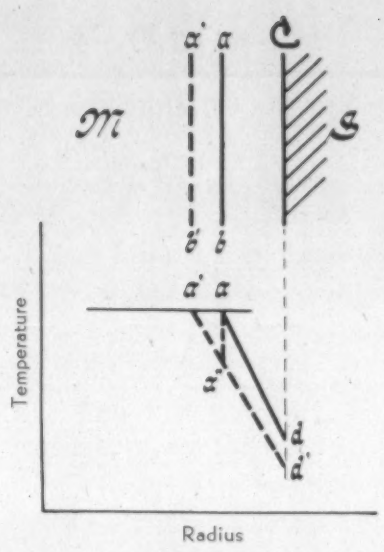


Fig. 5

Although gas separation is probably a primary cause, there are other possible causes for the generation of inner pressure. There is a possibility of the development of pressure in the inner part of an ingot owing to the shrinkage of its outer already crystallized part. Let S, Fig. 5, be the sand mold, M the cylindrical ingot in the process of solidification. Let part abcd be already solidified at a given moment. ab is then the border between the crystals and the liquid. Let this border pass on to a new position a′b′ separated by a length *dr* from ab during a time interval *dt*.

To simplify the calculation we assume, that the alloy crystallizes as a pure metal at constant temperature and not over a temperature range. Furthermore, we suppose, that the melt inside ab is at the melting temperature.

The temperature changes obtained under these conditions through the solidification of the layer aa′ are plotted in Fig. 5.

The temperature drop aa″ in the layer ab is greater than the fall dd′ on the surface, but for short periods of time we may assume, that they are equal to one another, thus aa″ = dd′.

Let Δv_s be the true contraction of volume ≦ by solidification, α the coefficient of linear temperature dilatation.

Knocking Out Ingots after Pigging Reverberatory Furnace in Background. (Courtesy Aluminum Company of America.)

Then the linear contraction at a by a cooling by $dT =$ aa″ degrees will be: $dr \cdot \alpha \cdot dT$

while the linear contraction through the solidification of the melt is $1/3\ dr \cdot \Delta v_s$

The condition for the start of shrinkage pressure is therefore $1/3\ dr \cdot \Delta v_s \leqq r \cdot \alpha \cdot dT$,

and thus for the temperature gradient in the solid metal

$$dT \leqq 1/3 \frac{\Delta v_s}{r\,\alpha}$$

Now in the case of bronze the solidification shrinkage is about 1%, thus $\Delta v_s = 0.01$ and $\alpha \sim 2.10^{-5}$ and we obtain Table 3 for the condition of shrinkage pressure.

Table 3.

Radius in cm.	Lowest value of $\frac{dT}{dr}$ in $\frac{°C}{cm.}$	Linear Crystallization velocity cm./sec.
2	80	0.12
5	35	0.05
10	17	0.02
20	8	0.01

To measure the temperature gradients actually occurring in a sand casting, we have poured bronze containing 15% Sn and 0.1% P into a sand mold 5 cm. in diameter and 10 cm. in height. The temperature of the melt was 1460° C. The temperature at the surface and in the center of the metal was measured by means of 2 thermocouples. As soon as solidification occurred at the surface, the temperature difference between the center and the surface was 80° C., and when it proceeded to the center it was still 55° C. The "sweating" began when the temperature in the center was 950° C. and when the difference was 50° C.

The temperature gradient during the process of sweating was far below 20° C./dm. and consequently much too low to develop a shrinkage pressure.

In the case of a low solidification rate, shrinkage can therefore not cause the mother liquid to sweat. The sole cause of the segregation is, in this case, gas evolution. We may call the inverse segregation of large sand castings "gas segregation."

Since there is evidently no other cause of the gas segregation, the technical means for avoiding it must prevent the gas from entering the metal during its melting. Another well known means is the raising of the cooling velocity, if this is possible. If the outer layer of the metal is cold enough at the time gas separation starts, the mother liquid must freeze in the interior of the ingot and cannot reach the surface.

In the case of rapid solidification inverse segregation is much harder to explain. In this case we are no longer able to indicate with any degree of certainty the true origin of the phenomenon. We can only show that some of the explanations suggested up to now are not adequate.

Although "sweating" can be avoided in bronze by increasing the cooling rate gas pressure cannot be the sole cause of inverse segregation in chill cast metals. The lack of segregation at intermediate cooling velocities between the very rapid and the very slow would make it difficult to explain why the gas pressure is without influence only at intermediate cooling velocities.

$$\frac{C_0 + SC_1}{1+S} \sim C_0 + S(C_1 - C_0)$$

Fig. 6. Capillary Segregation.

Moreover, as has been shown for aluminum, bronze and for brass, inverse segregation also occurs when the alloy is poured into a metal mold and poured out again before the core has crystallized. It is then found that the chill cast shell, if not too thin, shows an excess of tin or zinc as compared with the whole alloy as may be seen in Table 4.

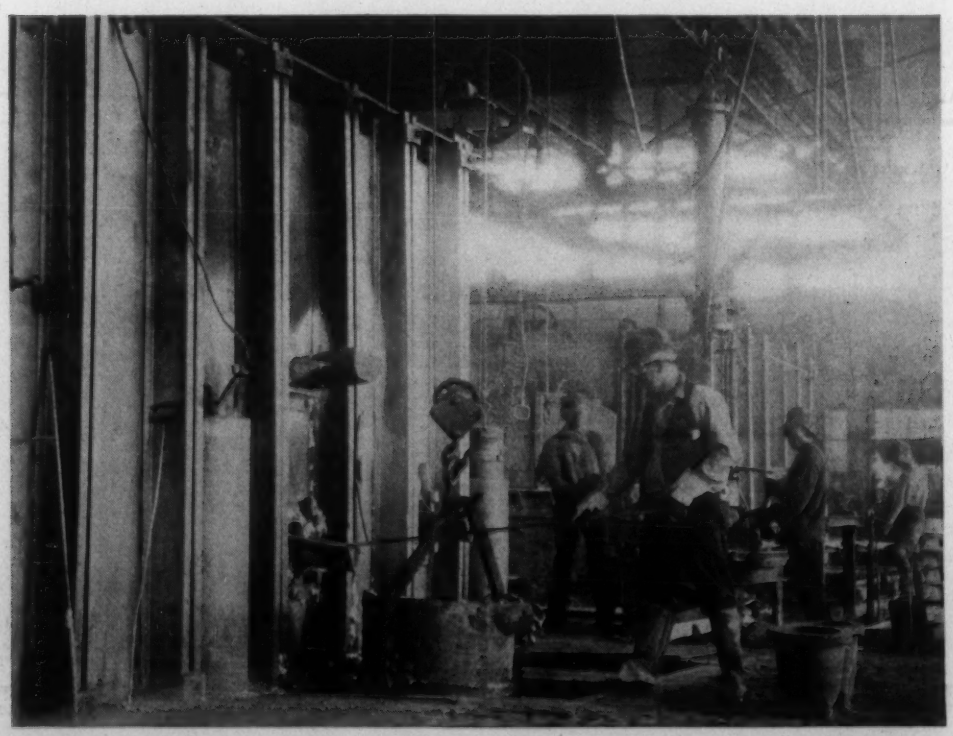
So long as there is still liquid in the mold a pressure cannot be developed. The fact, that the inverse segregation is nevertheless developed proves that it can occur without a transfer of the mother liquid by means of an inner pressure effect from the center to the outside.

Table 4.

Alloy	% Copper content of the whole alloy	% Copper content of the chill cast shell	Investigator
		Thickness 10-13 cm.	
Zn-Cu	12.42	11.85	Fraenkel and Goedecke
		Thickness 2-3 cm.	
	12.08	13.6	"
Al-Cu	11.57	12.27	Rosenhain and Archbutt

Although in the case of chill cast alloys the shrinkage pressure is probably of some influence, even in these cases the occurrence of voids in the interior of the castings shows that shrinkage pressure has therefore not been the only cause of inverse segregation.

The capillarity theory assumes, that the channels formed between the dendrites, crystallized at the outer surface of the ingot are being filled up with the mother liquid. This cannot explain the rate of inverse segregation actually found in some cases. Let ab (Fig. 6) be the wall of the metal mold and let the thick layer of the alloy abcd be crystallized. If the contraction of solidification in percent is Δv_2, the upper limit



Tapping 30,000 lb. Furnace. Metal will be Transferred in 800 lb. Ladles to Smaller Holding Furnaces.
(Courtesy Aluminum Company of America.)

of the weight of the liquid flowing into the outer dendritic cavities formed by crystallization in one gram of the alloy, will be $s = 0.01\,\Delta v_s$.

If the concentration of B (Fig. 6) in the alloy is c_o and its concentration in the part of the liquid solidifying last c_1 ($c_1 > c_o$), the concentration of B of the outer layer abcd, after the filling up of the channels, will be

$$\frac{c_o + sc_1}{1+s} = \frac{c_o(1+s) + s(c_1 - c_o)}{1+s} =$$
$$c_o + s(c_1 - c_o) = c_o + s\Delta c.$$

The filling up of the capillary channels begins as soon as solidification starts. The concentration of the melt in the channels rises during this time from c_o up to c_1. We may therefore assume, that we obtain the true value of c_o, if we assume it to be equal to the concentration of the melt when half of the alloy has been solidified, as shown in the equilibrium diagram. In Table 5 the changes of concentration calculated in this manner are compared with the test values. It will be seen, that in most cases the latter are far larger than the former, so that the "capillary flow" of the melt cannot be regarded as a general cause of inverse segregation.

Table 5.

Alloy	c_o	c_1	c_1-c_o	s	c as calculated	c as observed
Cu-Sn	.075	.125	.05	.01	.0005	.004
Al-Zn	.17	.30	.13	.06	.0078	.0083
Al-Cu	.012	.024	.012	.06	.00072	.0021
Al-Cu	.031	.071	.04	.06	.0024	.0033

One sees that while the "capillary action" can produce a certain amount of inverse segregation this amount in most cases is far below the amount determined experimentally. In opposition to the results obtained by C. Haase, the author has furthermore been able to show, that inverse segregation occurs in chill cast bismuth-antimony-alloys which crystallize with expansion. In this case, there is no formation of channels between the crystals into which the mother alloy might penetrate. Nevertheless inverse segregation occurs, as will be seen from Table 6.

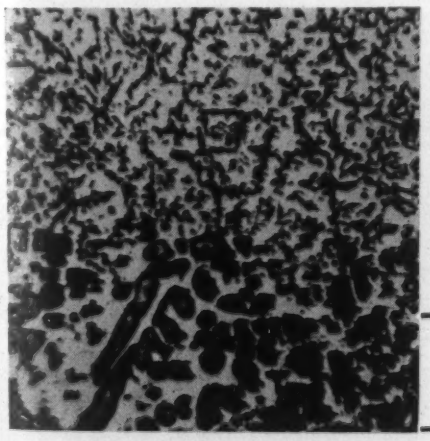
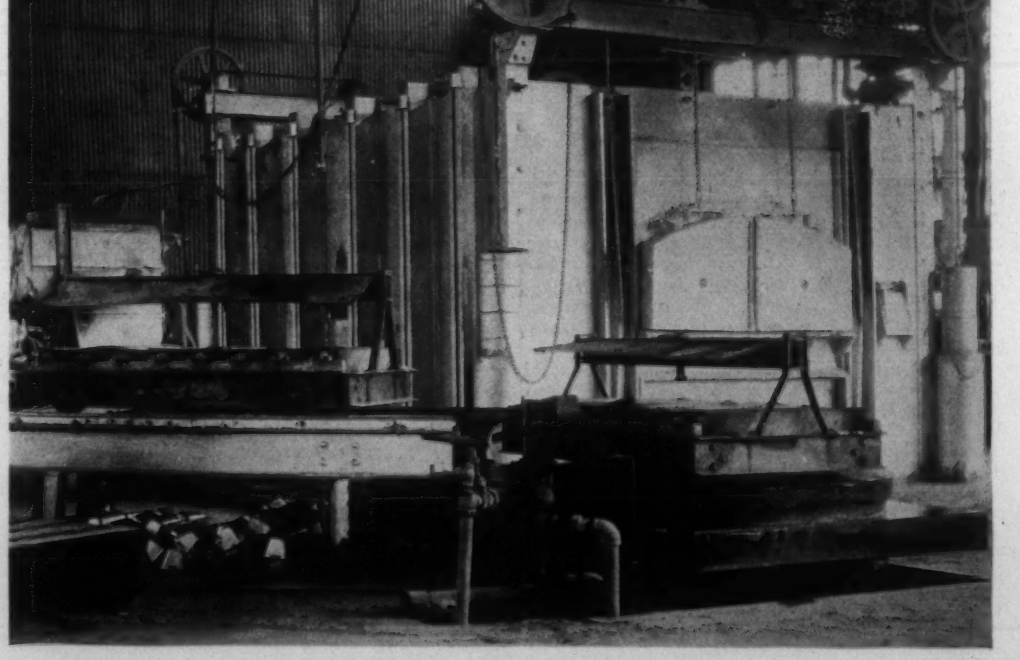


Fig. 7. 50% Ag-Cu. "Soaked" and Chilled. Upper Boundary. Silver: 51.2-49.0. Magnification 40X. (Watson.)

Table 6.

Method of Casting	Part of the ingot	Sb Content in %
Chill cast	Surface	22.68
	Core	23.38
Sand cast	Surface	21.43
	Core	23.17
Chill cast 20 mm.ϕ	Surface (18-20 mm.ϕ)	14.02
	16 – 18 mm.	14.33
	12 – 16 mm.	14.63
	6 – 12 mm.	14.96
	0 – 6 mm.	15.28



Ingot Mold Equipment used in Pigging Gas Fired Reverberatory Furnace.
(Courtesy Aluminum Company of America.)

There are therefore some other causes of inverse segregation in addition to the capillary flow of the melt.

The Ludwig-Soret-phenomenon has been advanced to explain inverse segregation. This phenomenon consists in a contration difference produced in a solution by a temperature gradient. As a phenomenon of diffusion it requires much time, and it seems quite improbable, that it should occur to any considerable amount in a metal poured into a metal mold, which crystallizes very quickly.

It has also been suggested, in explanation of inverse segregation, that the dendritic primary crystals move into the inner part of the melt.

J. H. Watson[1] has shown evidence of this movement in copper-silver alloys. Consider an alloy, in which primary crystals consist of copper of say 50% by weight. This alloy was poured into a crucible heated in a furnace and maintained there for 4 hours at a temperature between the liquidus and the solidus line. During this time large primary crystals of copper were formed, which had a certain tendency to rise to the top of the crucible owing to "density segregation," so that the silver concentration was lowered at the top to about 39% Ag. If the surface of the melt was then quenched by putting a cold metallic body upon it and by allowing the whole melt to crystallize from the top it was observed that the large primary crystals of copper were repulsed from the chilled surface of the alloy, thus forming an upper part freed from the large copper crystals. At an average distance of 0.25 cm. from the top, there was a boundary within which there were no large copper crystals. This boundary is clearly seen in Fig. 7.

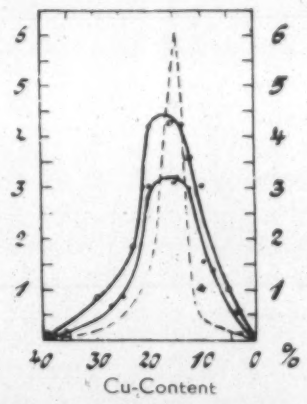


Fig. 8. Dilatation and Segregation of Zn-Cu Alloys.

Furthermore, Fraenkel and Goedecke[2] have shown that if a layer of an alloy is cast into a metal mold and the unsolidified portion then quickly poured out, the composition of this layer will depend on its thickness. If it is very thin, it shows normal segregation with 13.6% Cu in a copper-zinc alloy containing 12.42% Cu. But if it becomes

[1]Liquation or "Inverse Segregation" in the Silver-Copper Alloys. *Journal Institute of Metals*, Vol. 49, No. 2, 1932, pages 347-362.
[2]W. Fraenkel & W. Goedecke. *Zeitschrift für Metallkunde*, Vol. 21, 1929, pages 322-324.

thicker, its composition is found to be 11.85% Cu as compared with the mean value of 12.08% of the alloy. Now all the investigations on the inverse segregation of massive ingots show, that it is most pronounced at the surface of the ingots. It can therefore not be supposed, that the first layer of 2-3 cm. has a greater content in Zn than have the subsequent ones. It must be assumed, that the composition of the outer layer has been really changed during solidification.

Now what is the force which repulses the crystals from the wall of the mold? It seems to be a force accompanying crystallization. If the crystal growth is needle-like or dendritic, it is possible that growing crystals are repulsed from the wall. This idea has been suggested by the fact that there is a close relation between the measured increase in length at the end of the solidification, and the amount of inverse segregation in chill cast alloys. Fig. 8, taken from a paper by Jokibe shows this quite clearly. This elongation of the alloys is in contradiction with the specific weight of the solid metal, which is higher than in the liquid state: the crystallized body is porous. Now one might attribute this porosity to gas separation during solidification, and it is quite probable, that this explanation is correct within certain limits. But gas separation is not likely to depend on the concentration as given for the elongation in Fig. 8. Furthermore, the alloy containing 15% Cu 85% Zn shows an extension of quite another kind than the well-known extension of the melt at the end of the solidification process in bronze, as caused by gases and as discussed above. In the case of the former alloy the solidification proceeds at first in an entirely normal manner, including normal piping. Then, when the outer part has already been crystallized, the ingot begins to expand. Hence a considerable force is developed, which cannot be explained by a gas passing out of a liquid.

But if we assume that a force of crystallization is developed in this case, the phenomenon is comparable with the increase in volume owing to the hydration of calcium-oxide, although the true specific volume of calcium-hydroxide is smaller than the sum of the volumes of calcium oxide and water.

Let us now sum up as follows: While the gas segregation at low cooling rates may be explained by a single factor (the gas) other factors are also decisive for the inverse segregation in chill cast alloys. The shrinkage pressure apparently does not occur at ordinary cooling velocities. The influence of capillary flow of the mother liquid has not been verified experimentally, but it may doubtless be assumed when the alloy itself contracts in solidifying. But there are conditions for which the inverse segregation cannot be accounted for. These cases are explained by the assumption of a force of crystallization (or of diffusion immediately after crystallization), which repulses the needle-like crystals from the wall of the mold while the melt flows in to take their place.

In addition to inverse segregation, almost all the alloys, after rapid solidification, show a coring effect, originated by a lack of diffusion in the solid solutions. It is usually removed by homogenization at high temperatures where the concentration differences are diminished by diffusion.

Diffusion in many cases is a far more complicated process than we usually assume. Generally it is thought, that diffusion proceeds by the interchange of places of adjacent atoms in the space lattice. This simple mechanism of diffusion cannot explain the changes in volume and shape, which have been found after diffusion. Table 7 shows the volume changes due to diffusion in cast bismuth-antimony alloys which involve a grave coring effect.

Table 7.

Density changes in Sb-Bi alloys (25% Sb) after different annealings

	Annealing Temperature °C.	Density: Before Annealing	Annealed 10 hours	Annealed 48 hours	Annealed 72 hours
Cast in mold	300	8.822	7.919	7.498	7.254
Cast in mold	260	8.829	8.491	8.443	8.150
Frozen in crucible	300	8.824	8.362	8.423	8.353
Frozen in crucible	260	9.022	8.977	8.889	8.231
Cast in mold	300 (in absence of air)	8.818	8.143	——	

Lately we have found, that chill castings of bronze containing 10% Sn had a density of 8.755 in the cast condition, but after an annealing of 3 hours at 700° C., the density decreased to 8.709. In the micrographs, small holes are observed in centers where before the annealing the γ constituent had been present.

These anomalies of diffusion doubtless have a general significance; furthermore it is to be assumed, that, as a rule, both constituents of a solid solution travel with different velocities. This may not occur in the inner part of the undisturbed crystal, but along crystal boundaries or some weak points in crystals.

(To be continued in August)

Creep of Metals at High Temperatures

(Continued from page 98)

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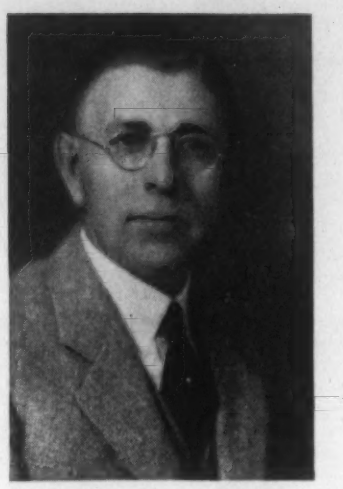
Priority Publication

MODERN METALLURGICAL ENGINEERING is becoming competitive, and engineers and research workers frequently justifiably desire to establish priority. METALS & ALLOYS therefore offers its columns for the publication of short notes describing work or ideas that are not sufficiently complete to warrant presentation before technical societies. The editor hopes that such brief notes will be sent in by a large number of readers, since they not only provide a convenient means of establishing priority but also permit publication in a less formal manner and with far more rapidity than a complete paper.

There, no doubt, have been many times, when you would like to have published an interesting observation, not worthy of a complete paper, in such a manner. Subjects particularly suitable for brief notes are properties of various alloys or a series of related alloys, unusual applications of known metals and alloys, uses of new alloys, laboratory technique, a modification or a new design of plant or laboratory equipment, etc., etc.—RICHARD RIMBACH.



Properties of The ALLOYS OF NICKEL with Tantalum*

by Eric Therkelsen**

ALTHOUGH a number of articles[1] have been written on the subject of tantalum, very little information has been published regarding its alloys. In 1913 a German patent[2] was granted to the firm of Siemens & Halske on "a process for improving the mechanical and chemical resistances of nickel by the addition of tantalum." Several references[3] are made in the literature to the nickel-tantalum alloys but none of them add to the information contained in the patent and some of them quote the patent verbatim.

This article describes a study of a series of nickel-tantalum alloys ranging in Ta content from 5% to 80%. The object of the study was to determine the mechanical, chemical and electrical properties of the malleable alloys up to 30% tantalum described in the Siemens & Halske patent, to examine the microstructure, and to construct as far as possible the constitutional diagram.

The metals used in the alloys were electrolytic nickel with less than 0.5% impurities including cobalt, and rolled tantalum sheet obtained from the Osram Lamp Works laboratories containing less than 0.5% impurities including columbium.

The alloys were melted in a vacuum furnace[4] having a heating element in the form of a spiral carbon tube which surrounded the crucible. Four kinds of crucibles were tried—hard porcelain, spinell, aluminum oxide and zirconium oxide, the

DESPITE all the man-years of metallurgical research, there still remain combinations of metals whose properties are almost unexplored, even when only two metals are concerned. This is particularly true in the case of some of the less common metals, such as Tantalum.

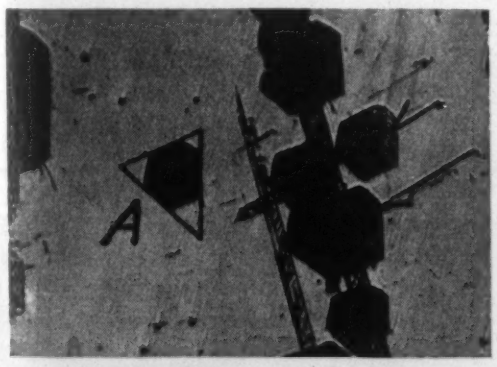
It is generally found that almost any alloy has properties that fit it for some use. Whether the alloy can compete with others depends on economic factors as well as technical ones, but there is always the possibility that some property may be so outstanding as to make even a costly alloy the proper one for some special use.

Even if the alloy serves no better than others, it is always a service to have its properties set forth, for then we have facts to go on instead of a gap where we know nothing. Every metallurgist looks at the periodic table now and then and wonders what hidden possibilities lie in the untried combinations.

These pioneer, gap-filling articles, replace mere speculation by facts.

*Abstract of a doctor dissertation, "Zur Kenntnis der Eigenschaften der Legierungen des Tantals mit Nickel." Technische Hochschule, Berlin, June 1932.

**Prof., Mechanical Engineering, Montana State College, Bozeman, Montana.



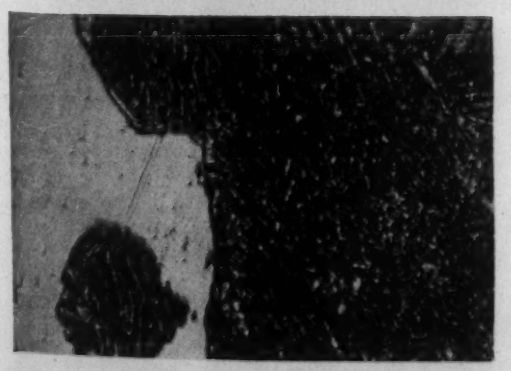


Fig. 1. Etching Pits in 27.3% Ta Alloy. Etchant HNO_3 50%. Magnification 100 X.
Fig. 2. 38.3% Ta Alloy. Etchant, Aqua Regia. Magnification 200 X
Fig. 3. 38.3% Ta Alloy. Etchant, Aqua Regia Magnification 525 X

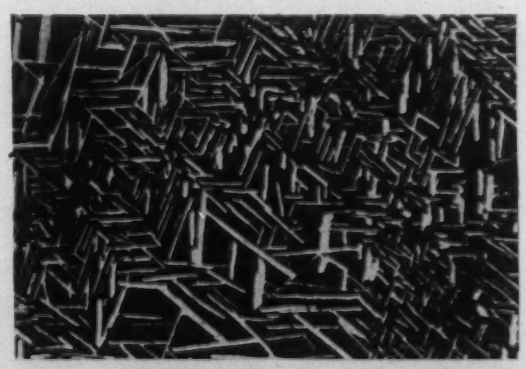
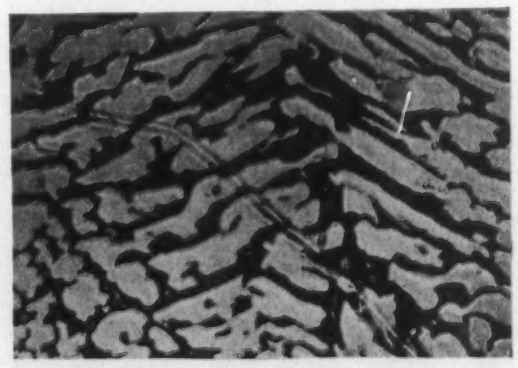


Fig. 4. 40.4% Ta Alloy. Etchant, Aqua Regia Magnification 50 X



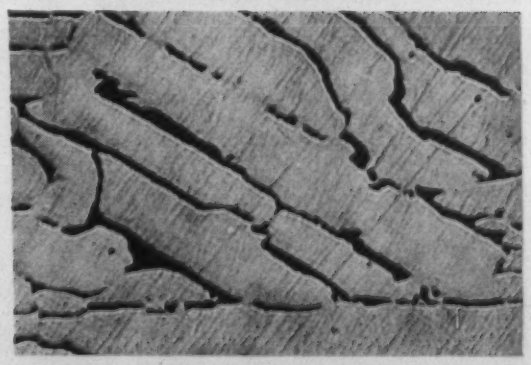


Fig. 5. 46.2% Ta Alloy. Etchant, Aqua Regia. Magnification 100 X
Fig. 6. 50.1% Ta Alloy. Etchant, Aqua Regia. Magnification 100 X
Fig. 7. 52% Ta Alloy. Etchant, HF. Magnification 100 X

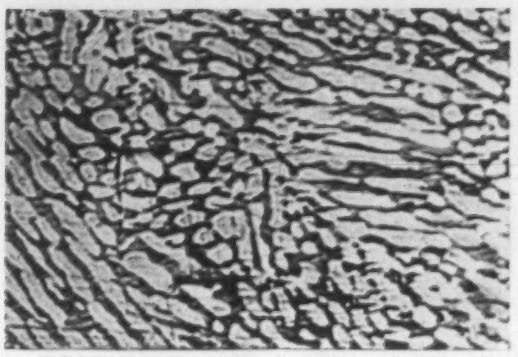


Fig. 8. 56.8 % Ta Alloy. Etchant, HF. Magnification 100 X

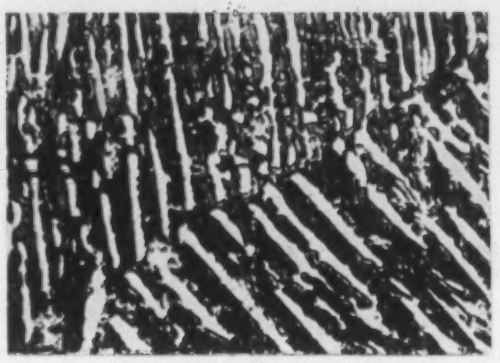


Fig. 9. 59.5 % Ta Alloy. Etchant, HF. Magnification 100 X

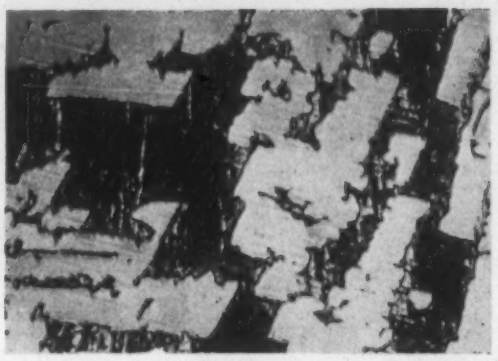


Fig. 10. 43.1 % Ta Alloy. Etchant, Aqua Regia. Magnification 100 X

zirconium oxide proving the best. The reguli had a weight of 30 to 40 grams. After the specimens had been polished and examined they were broken and analyzed to check the composition, the nickel content being determined as nickel dimethylglyoxim.

The etchant used for the alloys up to 32.6% Ta was HNO_3, for the alloys with 38.1% to 50.1% Ta, aqua regia, and for all others HF. The vapor from the last 2 etchants was more effective than the liquids. The structure of the nickel-tantalum alloys is various. Up to 32.6% Ta the specimens proved to be typical homogeneous solid solutions and all had a tendency to become very coarse grained, some of the reguli consisting of but 2 or 3 crystals. The etchant used for the solid solution alloys was 50% nitric acid. Fig. 1 shows an interesting example of etching pits which were started along scratches accidentally made by a glass rod. The pit at A with 3 sides projected shows the corner of a cube but with the extreme edges (the white corners) not eaten out by the etchant.

Two of the reguli with 38.1% and 40.4% Ta respectively possessed typical segregate structures as shown in Figs. 2 and 4. These alloys were evidently not in equilibrium as is shown by the gray patches between the segregate needles and the black borders around the needles. When the alloy of Fig. 2 was remelted and cooled much more slowly the darker background between the segregate crystals was found on higher magnification to have a lamellar structure as shown in Fig. 3. The further separation of the segregate crystals out of the solid solution at lower temperatures suggests that this alloy is fundamentally capable of being age hardened.

Figs. 5 and 6 illustrate the heterogeneous structures of 2 alloys with 46.2% and 50.1% Ta respectively. The dark background area of solid solution becomes smaller and the segregate area larger with increasing Ta content. Evidently the alloy of Fig. 6 is approaching the composition of the pure compound of the segregate. The white areas of the photographs of Figs. 2, 4, 5 and 6 were cut out and weighed and the proportion of white area plotted against the tantalum content of the alloys. The curve, extrapolated to 100% of white area gave 50.7% Ta as the composition of the segregate, which corresponds to the compound Ni_3Ta.

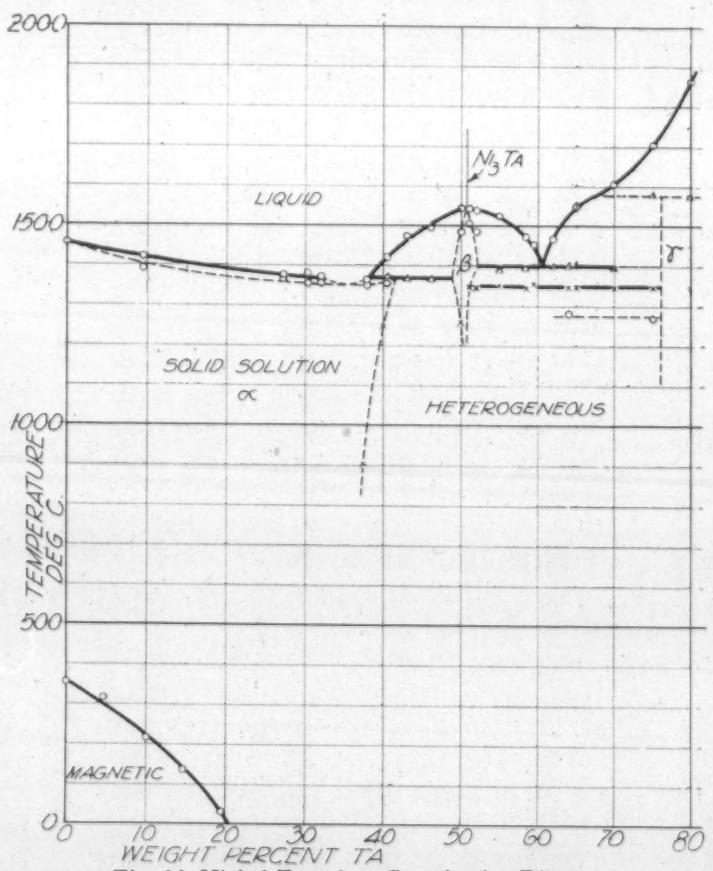


Fig. 14. Nickel-Tantalum Constitution Diagram

With more than 50.7% Ta the segregate area begins to diminish as shown in Figs. 7, 8 and 9 The dark background is not now the solid solution of the alloys with less than 50.7% Ta as is shown by the fact that if the etchant for the alloys above 50.7% Ta be aqua regia instead of hydrofluoric acid the segregate became the darkly etched portion. Also if hydrofluoric acid was used on the alloys with less than 50.7% Ta the segregate crystal became dark. This is illustrated by Figs. 10 and 11.

Plotting the segregate areas of the alloys above 50.7% Ta indicates that the segregated compound Ni_3Ta vanishes from the structure with 60.7% Ta.

The alloys with 62% Ta and above showed various new crystal forms.

A transformation in the solid state at 1350° C. (Fig. 14) made it difficult to interpret the mircrostructures. Fig. 12 shows 3 phases not in equilibrium, (1) the black background of the β crystal, (2) the long white needles which were evidently first segregated from the β crystal and (3) the roundish crystals formed during a transformation in the solid state. This structure was typical of the alloys containing more than 62% Ta.

These alloys with high tantalum content were very difficult to melt and showed evidence of contamination, due to oxidation

Fig. 11. 43.1 % Ta Alloy. Etchant HF. Magnification 100 X

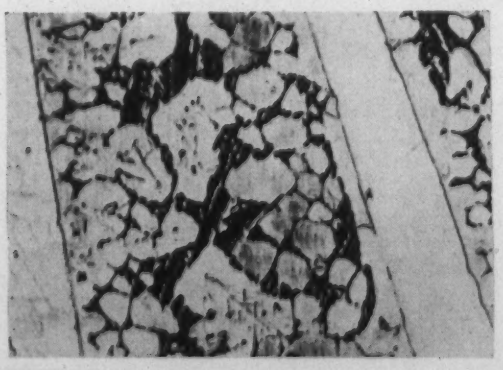


Fig. 12. 62 % Ta Alloy. Etchant, HF. Magnification 100 X

Fig. 13. 65 % Ta Alloy. Etchant, HF. Magnification 100 X

by the crucible, absorption of carbon vapor from the heating element and of gases in the furnace.

Temperature measurements for the thermal analyses were made by means of an optical pyrometer ("Pyropto," Hartmann & Braun). The vacuum furnace was provided with a quartz glass window 15 mm. in diameter. The pyrometer was calibrated from time to time by melting pure nickel, melting point 1453° C., and at this temperature the instrument read 25° too low, the largest variation in the 10 measurements being 5° C.

All of the alloys showed extraordinary undercooling, sometimes as much as 130° C. Even stirring with a porcelain tube would fail to start crystalization with 50° undercooling.

Fig. 14 shows the constitution diagram constructed as far as the data taken would warrant. Since maximum temperature attainable with the crucibles available was about 2000° C., while the melting point of tantalum[5] is 3027° C. the alloys above 80% Ta could not be

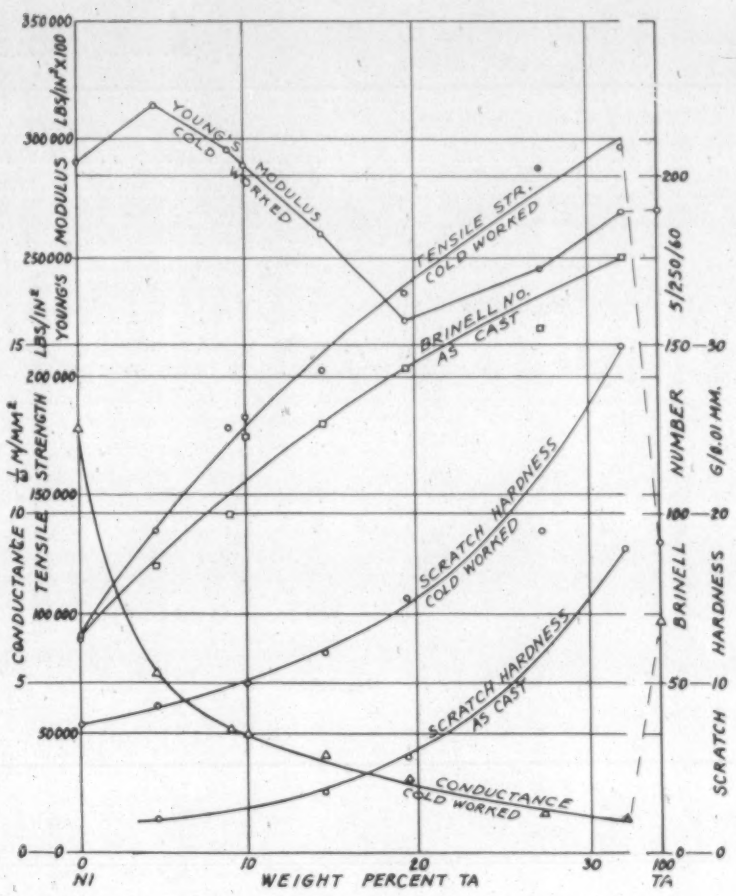


Fig. 15. Technical Properties of Malleable Ni-Ta Alloys.

modulus for the 32% alloy compares favorably with the values for steel. The scratch hardness of 30 for this alloy compares well with a value of 40 obtained by testing a safety razor blade. A scalpel made from this alloy made a very serviceable knife for desk use.

The electrical resistance of the cold drawn alloy with 9% Ta was tested at temperature t between 0° and 180° C., the resistance following the equation. $R = 0.262\ (1 + 2 \times 10^{-3}\ t + 1.6 \times 10^{-6}\ t^2)$ in which R = resistance in ohms per m. and mm.^{2}

Several thermal couples were made with the Ni-Ta alloys against Ni and Ta. The e.m.f. curves of these couples is shown in Fig. 16.

The corrosion resistance of the Ni-Ta alloys is of interest. The relative resistance to attack by various reagents was determined by the method of Guertler and Liepus[6] which consisted of placing the polished reguli in a glass dish, polished surface upward, and pouring the reagent over them all simultaneously. As each

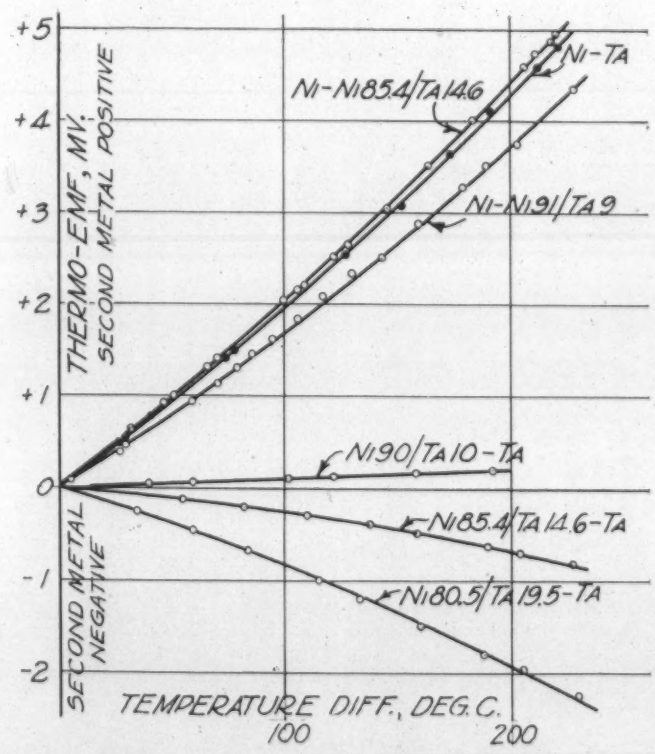


Fig. 16. Thermal e.m.f. of Ni-Ta Couples.

TIME REQUIRED TO PRODUCE A VISIBLE ETCH.
| = < 1 MINUTE • = ≥ 1 HOUR ◑ = > 24 HOURS
◦ = > 1 MINUTE ○ = > 8 HOURS ● = > 48 HOURS

PERCENT TA	PURE NI	4.6	9.0	10.4	14.6	19.5	27.3	30.3	32.6	100
AQUA REGIA	|	|	|	|	|	|	|	|	|	●
HNO_3 10 %	◦	◦	◦	◦	◦	◦	○	○	•	●
HNO_3 50 %	|	|	|	|	|	|	◦	◦	◦	●
HNO_3 CONC.	|	|	|	|	|	●	●	●	●	●
HCl 10 %	●	◦	◦	◦	◦	◦	●	●	●	●
HCl 50 %	◦	◦	◦	◦	◦	◦	●	●	●	●
HCl CONC.	◦	◦	◦	◦	◦	◦	•	•	•	●
H_2SO_4 10 %	◦	◦	◦	◦	◦	•	○	○	○	●
H_2SO_4 50 %	◦	◦	◦	◦	◦	◦	●	●	●	●
H_2SO_4 CONC	|	|	◦	◦	◦	•	●	●	●	●
CH_3COOH	◦	◦	◦	○	○	◑	●	●	●	●
$H_2C_4H_4O_6$	◦	◦	◦	◦	◦	◦	○	○	○	●
NH_4OH 5%	◦	◦	◦	◦	◦	◦	◦	◦	◦	●
NH_4OH 10%	◦	◦	◦	◦	◦	◦	◦	◦	◦	●
NaOH 10%	●	●	●	●	●	●	●	●	●	●
NaOH CONC.	●	●	●	●	●	●	●	●	●	●
DIST. H_2O, 100°C	○	•	○	•	•	•	●	●	●	●
CHLOR. H_2O	|	|	|	|	|	◦	●	●	●	●

Fig. 17. Summary of Corrosion Tests.

investigated. The compound Ni_3Ta, with a maximum on the liquidus curve at 1545° C. divides the diagram into 2 parts, the nickel side having the form of the Rooseboom type IV while the tantalum side is much more complicated. The relatively low melting point of the solid solution alloys is of great advantage in their practical production.

The alloys with up to 35% Ta are malleable and ductile. All the other alloys were exceedingly hard as measured by sawing and grinding but were brittle when hammered. Those with more than 60% Ta were so friable that some of them broke on being removed from the crucible. Only the malleable alloys were subjected to mechanical and chemical tests.

Fig. 15 shows the various technical properties of the malleable alloys plotted against tantalum content. The tests on the cold wrought specimens were made on wire rolled and drawn to 1.8 mm. diameter without annealing.

It will be noted that the tensile strength and Young's

specimen showed a distinct etch it was removed to clear water and the time noted and afterward checked by observation under the microscope. After each trial each specimen was repolished with Al_2O_3 and cleaned only mechanically with cotton. Fig. 17 gives graphically the results of the corrosion tests. All of the alloys oxidized on being heated in the air to about 350° C.

SUMMARY

1. Nickel and tantalum form a continuous series of solid solutions between 0% and about 36% Ta and form at least one compound, Ni_3Ta.

2. The liquidus temperatures of the alloys between 0 and 36% Ta lie below that of pure Ni. The compound Ni_3Ta melts at a maximum point on the liquidus.

3. All the solid solution alloys are malleable with strength and hardness increasing with Ta content. An improvable alloy with about 36-37% Ta is fundamentally possible.

4. The thermo e.m.f. of a couple consisting of Ni and Ni-Ta alloy (9 to 15% Ta) is about 2 mv. per 100° C. temperature difference.

5. The corrosion resistance of the solid solution alloys is very good especially in the region of 12½ atomic % Ta.

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[2]Deutsches Reich Patent 277242, Mar. 4, 1931.

[3]Nickel-Tantalum Alloys. *Iron Age,* Vol. 96, 1915, page 467.

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[4]This furnace is described in *Zeitschrift für Metallkunde,* Vol. 20, 1928, page 271.

[5]The most commonly used temperature for the melting point is 2850° C. as given by Pirani, *Zeitschrift für Elektrochemie,* Vol. 17, 1911, page 908. A more recent and probably more reliable value is 3027° C. as given by A. G. Worthing, *Physical Review,* Vol. 28, 1926, page 174, according to a personal statement to the author by Professor Pirani.

[6]*Zeitschrift für Metallkunde,* Vol. 17, 1925, page 310.

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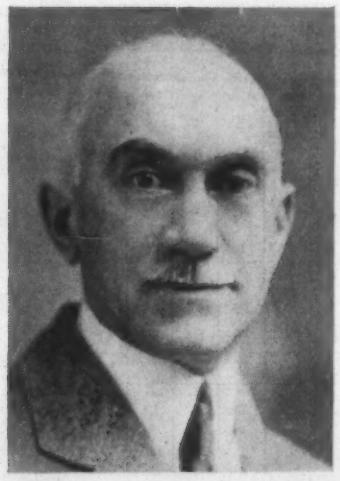

The American Foundrymen's Association has awarded the Joseph S. Seaman medal, for service to the foundry industry, to **G. H. Clamer,** of the Ajax Metal Company. Mr. Clamer was a pioneer in applying technical control to the secondary non-ferrous metal industry. Through his interest and support, the Ajax-Wyatt and Ajax-Northrup electric induction furnaces were developed and commercialized. Mr. Clamor is prominent in the movement to simplify brass and bronze alloy specifications and eliminate unnecessary "mixtures." He takes an active part in many technical societies, having been especially prominent in the A.F.A. On June 5th, Ursinus College, Collegeville, Pa., conferred the Honorary Degree of Doctor of Science on Mr. Clamer.

◈

The honorary degree of Doctor of Science, has been conferred upon **H. A. Schwartz,** Director of Research, National Malleable & Steel Castings Co. and a member of the Editorial Advisory Board of METALS & ALLOYS by his alma mater, Rose Polytechnic Institute.

◈

Ludlum Steel Company, Watervliet, New York, announces that **P. E. Floyd** has been appointed District Sales Manager in charge of the Chicago territory.

◈

Dr. E. A. Rykenboer has been appointed general manager of The R. & H. Chemicals Department of the du Pont Company to succeed **C. K. Davis,** who was recently elected president and general manager of the Remington Arms Company, Inc.; and **Milton Kutz** has been appointed assistant general manager of the department to succeed Dr. Rykenboer.

◈

Effective May 20, 1933, the Buffalo District Sales Office of Republic Steel Corporation will be moved to 475 Abbott Road, Buffalo, N. Y. **Thos. B. Davies,** District Sales Manager, and his present staff will continue in charge at the new location.

Spectroscopic Conference at M.I.T.

A five-day Spectroscopic conference is to be held at Massachusetts Institute of Technology, July 17-22, during a summer research gathering of spectroscopists, with meetings in the mornings only. The daily programs, in chronological order are Quantitative Spectroscopic Analysis of Materials, Biological and Photo-chemical Applications of Spectroscopy, Analysis of Complex Spectra, Spectroscopy and Atomic Structure.

From three to five papers are listed for each session, with more probably to be presented. Each set of papers is to be followed by "extended discussion of the present status and future possibilities of each branch of spectroscopy considered," which, plus the program, sounds like a long morning's work.

The only session likely to be of interest to metallurgists and chemists concerned with metallurgical analysis is the first one. The program for this follows:

C. C. Nitchie, Bausch and Lomb Optical Co. Some Practical Applications of the Spectrograph.

W. F. Meggers, Bureau of Standards. The Physical Basis for Quantitative Spectrochemical Analysis.

F. Twyman, Adam Hilger, Ltd. The Role of the Instrument Maker in Spectrum Analysis.

C. E. K. Mees, Eastman Kodak Co. Photographic Plates for Spectroscopy.

Kelvin Burns, Allegheny Observatory. Leader of Discussion on Spectro-chemical Analysis.

Advanced Welding Course in Cleveland

July 10 is the date set for another Advanced Welding Course to be given by the John Huntington Polytechnic Institute, Cleveland, Ohio.

The course, now being repeated due to the success of previous ones, is designed to give engineers and experienced operators an opportunity for a week's intensive study from both the practical and theoretical standpoints.

Complete information on the course may be obtained from E. W. P. Smith, Welding Engineering Department, John Huntington Polytechnic Institute, Cleveland, Ohio.

A Correction

Referring to the caption of Fig. 1 of "The Nature of the Solid Solution of Aluminum in Silver," by Dr. Charles S. Barrett, the figures 3.01% and 5.04% should be interchanged to agree with the diffraction patterns as printed.

New Officers of A.S.T.M.

At the annual meeting in Chicago the following officers' election was announced:

President: T. R. Lawson, Head, Dept. of Civil Engineering, Rensselaer Polytechnic Institute.

Vice-Pres.: Hermann von Schrenk, Consulting Timber Engineer.

Members of Executive Committee:

Frank A. Barbour, Consulting Hydraulic Engineer.

A. C. Fieldner, Chief Engineer, U. S. Bureau of Mines.

C. N. Forrest, Barber Asphalt Co.

J. C. Pearson, Director of Research, Lehigh Portland Cement Co.

A. E. White, Professor of Metallurgical Engineering, University of Michigan.

E. F. Houghton & Company Promotions

The Board of Directors of E. F. Houghton and Company, Philadelphia, Pa., announces the following changes and promotions in executive personnel:

Mr. Louis E. Murphy, president since 1929, continues in that capacity.

Major A. E. Carpenter, First Vice-President and Treasurer, resigned the latter office and was elected General Manager.

Mr. George W. Pressell, Second Vice-President and Secretary, resigned the secretaryship and was elected Assistant General Manager and Director of Sales.

Dr. R. H. Patch, Director of Plants, has been elected Treasurer.

Mr. A. E. Carpenter II, assistant to General Sales Manager, was elected Secretary.

The Board of Directors also announce the appointment of Mr. George S. Rogers, formerly Assistant General Sales Manager in charge of the St. Louis and Chicago offices, as General Sales Manager with headquarters in Philadelphia.

Manganese Steel Forge Sold

The entire business and assets of the Audubon Wire Cloth Company, Inc., wire cloth and wire products manufacturers of Audubon, N. J., were acquired by the Manganese Steel Forge Co., of Philadelphia, Pa., on May 1, 1933.

The business will be conducted by the Audubon Wire Cloth Corporation, a new organization and wholly owned subsidiary of the Manganese Steel Forge Co. The Officers of the new Corporation are: L. W. Jones, Pres.; L. W. Jones, Jr., V. P. & Treas.; A. W. Zackey, Secy.